



No. 11-149158- -00000-0000

Fecha: 2011-11-03 13:59:03 Dep. 2020 DIR.NUEVASCR
Tra. 11 PATENTEIYII Eva: 378 FASENACIONALI
Act. 411 PRESENTACION Folios: 141

Act. 411 PRESENTACION Folios: 138

Industria y Comercio

SUPERINTENDENCIA

DELEGATURA PROPIEDAD INDUSTRIAL

SOLICITUD DE PATENTE DE INVENCION
PCT

EXPEDIENTE No _____

TITULO MÉTODOS PARA EL USO DE ADITIVOS
DE PÉRDIDA DE FLUIDO QUE CONTIENEN
MICROGELES

CLASIFICACION INTERNACIONAL _____

SOLICITANTE HALLIBURTON ENERGY SERVICES,
INC

DOMICILIO Duncan, Oklahoma, Estados Unidos de
América

APODERADO DARIO CARDENAS NAVAS

BOGOTA D.C. _____



No. 11-149158 - 00000-0000

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Act. 411 PRESENTACIÓN Folios: 138

**FORMULARIO UNICO DE SOLICITUD DE PATENTE
PCT
ENTRADA EN FASE NACIONAL**

①

SOLICITUD DE:



Patente de Invención



Patente de Modelo de Utilidad



Capítulo I



Capítulo II

②

SOLICITANTE

Nombre: **HALLIBURTON ENERGY SERVICES, INC**

Dirección: PO Box 1431, Duncan, Oklahoma 73536

Nacionalidad o Domicilio: Duncan, Oklahoma, Estados Unidos de América

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IDENTIFICACION



NIT



NIT



Otro



Otro

Cuál
Número

③

REPRESENTANTE
O APODERADO

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IDENTIFICACION



NIT



NIT



Otro



Otro

Cual
Número 17066629 TP 1250
BTA C.S.J.

④

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2. Texas, Estados Unidos de América

⑤ PCT (86)

Solicitud Internacional No. WO 2010/112866 A1 Fecha 07.10.2010

Publicación Internacional No. PCT/GB2010/000657 Fecha 01.04.2010

⑥

Título(54): **MÉTODOS PARA EL USO DE ADITIVOS DE PÉRDIDA DE FLUIDO QUE
CONTIENEN MICROGELES**

⑦

Clasificación Internacional (51):

⑧

Prioridad

(33)País de Origen

(32)Fecha

(31)N° de Solicitud

US

03.04.2009

12/418,323

SI ☒ NO ☐

⑨

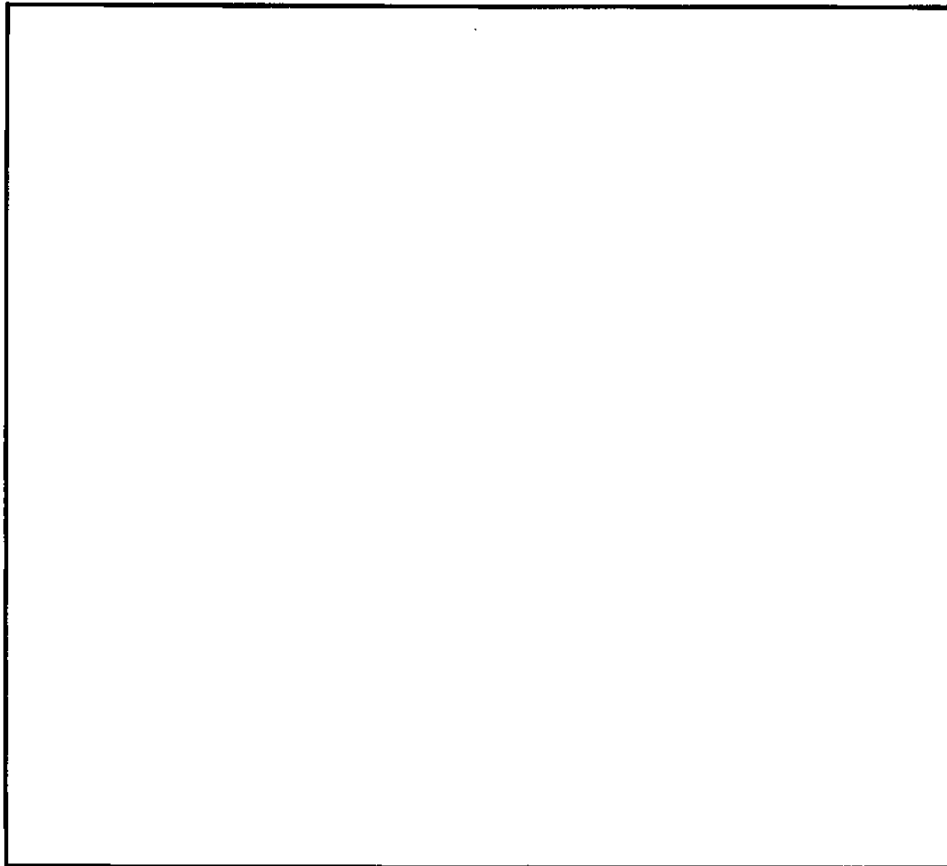
Comprobante de Pago No.: 11-102768

Fecha 20.10.2011

10 ANEXOS

- ☒ Comprobante de pago de la tasa de presentación de la solicitud.
- ☐ Documento que acredita la existencia y representación legal cuando el solicitante sea persona jurídica.
- ☐ Poderes, si fuere el caso
- ☐ Documento de cesión del inventor al solicitante o a su causante.
- ☒ Traducción de la solicitud internacional al castellano (Resumen, Resumen, descripción, reivindicaciones, dibujos)
- ☐ Copia del examen preliminar internacional efectuado por la Autoridad Internacional de Examen Preliminar (IPEA)
- ☐ Traducción del examen preliminar internacional efectuado por la IPEA.
- ☐ De ser el caso, copia del contrato de acceso.
- ☐ De ser el caso, documento que acredite la licencia o autorización de uso de conocimientos tradicionales de las comunidades indígenas.
- ☐ De ser el caso, certificado de depósito del material biológico.
- ☐ Arte final 12x12.
- ☐ De ser el caso, información sobre otras solicitudes de patente o títulos obtenidos en el extranjero por el mismo titular o su causante, relacionadas parcial o totalmente con la invención de esta solicitud.
- ☒ Recibo Reivindicaciones adicionales No 11-0099482 /11-0099457
- ☒ Prioridad
<http://www.wipo.int/patentscope/search/en/detail.jsf;jsessionid=53508A00BC13B5E7B50732805326F4CC.wapp1?docId=WO2010112866&recNum=1&tab=PCTDocuments&maxRec=&office=&prevFilter=&sortOption=&queryString=>

11 FIGURA CARACTERISTICA



12

Solicito la Concesión de la patente

NOMBRE: **DARIO CARDENAS NAVAS**

FIRMA:

C.C. 17066629 BTA TP. 1250 CSJ

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
NIT : 800.176.089-2
- / -



RECIBO DE CAJA

No. 11 - 102768

Bogotá D.C., Octubre 20 de 2011 - 15:29:01

RECIBIDO DE : CARDENAS & CARDENAS ABOGADOS PI LTDA NI 830.083.491

*** Soporte del Pago ***

TIPO PAGO	BANCO	CUENTA	No. PAGO	FECHA PAGO	VR. PAGO
CONSIGNACION	BANCO DE BOGOTA	062754387	427474460	20/10/2011	9.764.000.00

*** Conceptos Pagados ***

CANT.	RENTISTICO	CONCEPTO	Vr. UNIDITARIO	Vr. CONCEPTO
1	50005-01-01 SOLICITUDES	1 TRAMITES DE SOL. DE PATENTE DE INVENCIÓN	571.000.00	571.000.00
				\$571.000.00

SON: **QUINIENTOS SETENTA Y UN MIL PESOS MONEDA CORRIENTE***

Responsable: _____
Recibo de Caja Aplicado al Expediente No. _____

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

No. 11-149158-00000-0000

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ABOGADOS

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
NIT : 800.176.089-2
- / -

 Industria y Comercio SUPERINTENDENCIA	RECIBO DE CAJA	No. 11 - 0099482
		Bogotá D.C., Octubre 10 de 2011 - 15:04:00

RECIBIDO DE : CARDENAS & CARDENAS ABOGADOS PI LTDA					NI 830.083.491	
*** Soporte del Pago ***						
TIPO PAGO	BANCO	CUENTA	No. PAGO	FECHA PAGO	VR. PAGO	
CONSIGNACION	BANCO DE BOGOTA	062754387	186851383	10/10/2011	14.910.400.00	

*** Conceptos Pagados ***				
CANT.	RENTISTICO	CONCEPTO	Vr. UNDITARIO	Vr. CONCEPTO
16 50005-01-01	SOLICITUDES	1607 REIVINDICACION UNITARIA ADICIONAL A LAS 10 INICIALES	27.000.00	432.000.00
				\$432.000.00

SON: **CUATROCIENTOS TREINTA Y DOS MIL PESOS MONEDA CORRIENTE***

Responsable: _____

Recibo de Caja Aplicado al Expediente No. _____

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
No. 11-149158-00000-0000
Fecha: 2011-11-03 13:59:03 Dep. 2020 DIR NUEVASCR
Tra. 11 PATENTEIYII Eve: 378 FASENACIONALI
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CARDENAS & CARDENAS
ABOGADOS

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
NIT : 800.176.089-2
- / -



RECIBO DE CAJA

No. 11 - 0099457

Bogotá D.C., Octubre 10 de 2011 - 15:04:00

RECIBIDO DE : CARDENAS & CARDENAS ABOGADOS PI LTDA NI 830.083.491

*** Soporte del Pago ***

TIPO PAGO	BANCO	CUENTA	No. PAGO	FECHA PAGO	VR. PAGO
CONSIGNACION	BANCO DE BOGOTA	062754387	186851383	10/10/2011	14.910.400.00

*** Conceptos Pagados ***

CANT.	RENTISTICO	CONCEPTO	VR. UNDITARIO	VR. CONCEPTO
1	50005-01-09 OTROS SERVICIOS DE PROPIEDAD INDUSTRIAL	21 CERTIFICACIONES	14.000.00	14.000.00
				\$14.000.00

SON: **CATORCE MIL PESOS MONEDA CORRIENTE**

Responsable: _____

Recibo de Caja Aplicado al Expediente No. _____

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

No. 11-149158 - 00000-0000

Fecha: 2011-11-03 13:59:03 Dep. 2020 DIR.NUEVASCR
Tra. 11 PATENTEIYII Eve: 378 FASENACIONALI
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CARDENAS & CARDENAS
ABOGADOS

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
NIT : 800.176.089-2
- / -

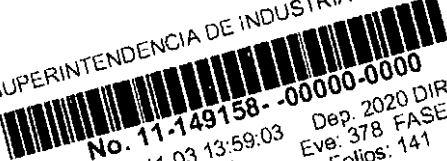
 Industria y Comercio SUPERINTENDENCIA	RECIBO DE CAJA	No. 11 - 0099458
		Bogotá D.C., Octubre 10 de 2011 - 15:04:00

RECIBIDO DE : CARDENAS & CARDENAS ABOGADOS PI LTDA					NI 830.083.491	
*** Soporte del Pago ***						
TIPO PAGO	BANCO	CUENTA	No. PAGO	FECHA PAGO	VR. PAGO	
CONSIGNACION	BANCO DE BOGOTA	062754387	186851383	10/10/2011	14.910.400.00	

*** Conceptos Pagados ***						
CANT.	RENTISTICO	CONCEPTO	Vr. UNDITARIO	Vr. CONCEPTO		
1	50005-01-09 OTROS SERVICIOS DE PROPIEDAD INDUSTRIAL	21 CERTIFICACIONES	14.000.00	14.000.00		
				\$14.000.00		
			=====			

SON: **CATORCE MIL PESOS MONEDA CORRIENTE**

Responsable: _____
****_
Recibo de Caja Aplicado al Expediente No. _____

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

No. 11-149158-00000-0000
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~~Act. 411 PRESENTACION~~ Folios: 138

CARDENAS & CARDENAS
ABOGADOS



Industria y Comercio

SUPERINTENDENCIA

(21) N° de solicitud:

(22) Fecha de solicitud:

(51) Clasificación Internacional:

(71) Solicitante(s) **HALLIBURTON ENERGY SERVICES, INC**

(72) Inventor(es)

Ryan G. Ezell, Jun Jim Wu

(74) Apoderado: **DARIO CARDENAS NAVAS**

(30) Prioridad	(31) <i>No. Prioridad</i>	32) <i>Fecha</i>	(33) <i>País</i>
	12/418,323	03.04.2009	US

(85) Fecha inicio fase nacional: 03.11.2011

(86) Datos relativos a la presentación de la solicitud PCT

Fecha de presentación de la solicitud: 01.04.2010

No. de solicitud *PCT/GB2010/000657*

(54) Título **MÉTODOS PARA EL USO DE ADITIVOS DE PÉRDIDA DE FLUIDO QUE CONTIENEN MICROGELES**

5

(21) N° de solicitud:				(22) Fecha de solicitud:			
(51) Clasificación Internacional :							
(71) Solicitante(s) HALLIBURTON ENERGY SERVICES, INC							
(72) Inventor(es) Anthony Curtis Philip							
(74) Apoderado: DARIO CARDENAS NAVAS							
(30) Prioridad		(31) <i>No. Prioridad</i>		(32) <i>Fecha</i>		(33) <i>País</i>	
		12/418,323		03.04.2009		US	
(85) Fecha límite inicio fase nacional: 03.11.2011				(87) Publicación internacional:			
(86) Datos relativos a la presentación de la solíc. PCT				Fecha: 07.10.2010			
Fecha de presentación de la solicitud: 01.04.2010				No. Publicación WO2010/112866 A1			
No. de solicitud <i>PCT/GB2010/000657</i>							

(54) Título: **MÉTODOS PARA EL USO DE ADITIVOS DE PÉRDIDA DE FLUIDO QUE CONTIENEN MICROGELES**

(57) Resumen:

Se proporcionan métodos y fluidos que incluyen pero no están limitados a un lodo de perforación que contiene un fluido base acuoso y un aditivo para controlar la pérdida de fluido que contiene por lo menos un microgel polimérico; y un método que comprende: proporcionar un fluido de tratamiento con base acuosa que contiene un aditivo para controlar la pérdida de fluido que comprende por lo menos un microgel polimérico; colocar al fluido de tratamiento con base acuosa en una formación subterránea a través de un orificio de pozo que penetra a esta última; permitir que el aditivo para el control de pérdida de fluido quede incorporado en una torta de filtro situada sobre una superficie dentro de la formación subterránea; permitir que la torta de filtro sea degradada y producir hidrocarburos desde la formación. Se proporcionan también métodos adicionales.

6

(21) N° de solicitud:				(51) Int Cl:	
22) Fecha de solicitud:				(71) Solicitante(s) HALLIBURTON ENERGY SERVICES, INC	
(30) Prioridad	(31) No. Prioridad	32) Fecha	(33) País	(72) Inventor(es) Ryan G. Ezell, Jun Jim Wu	
	12/418,323	03.04.2009	US	(74) Apoderado: DARIO CARDENAS NAVAS	
(54) Título: MÉTODOS PARA EL USO DE ADITIVOS DE PÉRDIDA DE FLUIDO QUE CONTIENEN MICROGELES					

Resumen:

Se proporcionan métodos y fluidos que incluyen pero no están limitados a un lodo de perforación que contiene un fluido base acuoso y un aditivo para controlar la pérdida de fluido que contiene por lo menos un microgel polimérico; y un método que comprende: proporcionar un fluido de tratamiento con base acuosa que contiene un aditivo para controlar la pérdida de fluido que comprende por lo menos un microgel polimérico; colocar al fluido de tratamiento con base acuosa en una formación subterránea a través de un orificio de pozo que penetra a esta última; permitir que el aditivo para el control de pérdida de fluido quede incorporado en una torta de filtro situada sobre una superficie dentro de la formación subterránea; permitir que la torta de filtro sea degradada y producir hidrocarburos desde la formación. Se proporcionan también métodos adicionales.

**MÉTODOS PARA EL USO DE ADITIVOS DE PÉRDIDA DE FLUIDO QUE
CONTIENEN MICROGELES**

La presente invención se refiere a métodos para usar aditivos para el control de pérdida de fluido. En forma más específica, por lo menos en algunas formas, la presente invención se refiere al uso de aditivos para el control de pérdida de fluido en fluidos de perforación y servicio que contienen microgeles para operaciones subterráneas.

Cuando se perfora orificios de pozo en formaciones productoras, se utiliza fluidos de perforación que reducirán el daño a la permeabilidad de tales formaciones y a su estabilidad, para producir hidrocarburos. Se utiliza fluidos de servicio cuando se realiza operaciones de terminación en formaciones productoras y cuando se realiza operaciones de reacondicionamiento en las formaciones. Los fluidos de perforación y servicio depositan una capa de partículas conocidas como "torta de filtro" sobre las paredes de los orificios de pozo dentro de las formaciones productoras. Se cree que la torta de filtro ayuda a evitar que los fluidos de perforación y de servicio se pierdan en la formación y evita que los sólidos entren en las porosidades de la roca. Después de la terminación y antes de iniciar la producción, se descompone generalmente a la torta de filtro o se deja

8

que se descomponga a fin de permitir que el producto fluya hacia el orificio de pozo para la producción. Es importante la descomposición de la torta de filtro para conservar la conectividad del orificio de pozo y la permeabilidad natural de la roca del yacimiento. Si no se descompone o se permite que se descomponga, la torta de filtro podría presentar un impedimento para la producción, entre otras cosas, mediante la alteración de la permeabilidad del yacimiento. Una vez que se ha disminuido la permeabilidad del yacimiento, raras veces es posible devolverlo a su condición original. Esto se podría distinguir de la función de los aditivos (denominados algunas veces en el arte "modificadores de permeabilidad relativa" o "RPMs") que se utilizan frecuentemente en fluidos de conformación o fracturación para sellar de manera permanente las entradas de agua hacia las áreas del yacimiento de hidrocarburo que rodean a un orificio de pozo.

Los fluidos de perforación y servicio (tales como fluidos de perforación) pueden contener aditivos para el control de pérdida de fluido a fin de ayudar a evitar que tales fluidos se pierdan hacia las formaciones. Los fluidos de perforación son cualesquiera de cierto número de fluidos y mezclas de fluidos y sólidos (como suspensiones sólidas, mezclas y emulsiones de líquidos, gases y sólidos) utilizados en operaciones para perforar

orificios de sondeo en la tierra. Se ha intentado en muchas formas la clasificación de los fluidos de perforación, produciendo frecuentemente mayor confusión que percepción. Un esquema de clasificación, ofrecido aquí, se basa solamente en la composición de lodo destacando al componente que define claramente la función y rendimiento del fluido: (1) basado en agua, (2) basado en aceite y (3) gaseoso (neumático). Cada categoría tiene una variedad de subcategorías que se sobreponen entre si en forma considerable. Idealmente, un lodo de perforación no es perjudicial para la formación, lo cual significa que no deja partículas, finos, etc., que tengan un impacto negativo en la permeabilidad de la formación.

Un lodo de perforación es un fluido destinado a perforar a través de la sección de yacimiento de un orificio de pozo en una formación subterránea. Las razones para usar un fluido especialmente diseñado incluyen pero no están necesariamente limitadas a: (1) perforar a la zona de yacimiento en forma satisfactoria, frecuentemente un orificio de drenaje largo y horizontal; (2) reducir los daños y aumentar la producción de las zonas expuestas; y (3) facilitar la terminación deseada del pozo, la cual puede incluir procedimientos complicados. Un lodo de perforación se asemeja frecuentemente a un fluido de terminación que puede

comprender una salmuera, posiblemente agentes para formar enlaces y/ polímeros.

El término "lodo de perforación", tal como se emplea aquí, se refiere genéricamente tanto a lodos de perforación como a fluidos de perforación a menos que se especifique de otro modo.

Otros tipos de fluidos de tratamiento que pueden utilizar materiales para el control de pérdida de fluido incluyen pero no están limitados a pastillas (tales como pastillas de tamiz interno), que son fluidos con una cantidad relativamente pequeña (por ejemplo, menos de 200 barriles) de una mezcla especial de lodo de perforación para realizar una tarea específica que el lodo de perforación del tipo regular no puede realizar. Ejemplos incluyen pastillas de alta viscosidad para ayudar a elevar a los fragmentos fuera de un orificio de pozo vertical; pastillas de agua dulce para disolver formaciones de sal invasoras, pastillas para liberación de tubería destinadas a destruir a la torta de filtro y aliviar las fuerzas de adhesión diferencial, así como pastillas de material de circulación perdida para taponar una zona de robo. Otro ejemplo es una pastilla tamiz que puede ser útil conjuntamente con una operación de relleno con grava.

Ejemplos de aditivos convencionales para controlar la pérdida de fluidos de tratamiento basados en agua,

incluyen polímeros solubles en agua no iónica tales como almidones, almidones derivados, gomas, gomas derivadas y materiales celulósicos. Los aditivos de pérdida de fluido incluyen almidones que varían frecuentemente en la proporción de contenido de amilosa a amilopectina y pueden ser modificados o no con un agente de degradación tal como epiclorohidrina. Del mismo modo, los almidones naturales no pueden ser uniformes en términos de calidad y efectividad. Estos almidones degradados frecuentemente no tienen estabilidad térmica a temperaturas hasta de más o menos 250°C y a temperaturas por encima de 250°F pueden ser utilizados de manera efectiva solamente mediante el aumento de la carga del fluido de tratamiento con el almidón degradado, reabasteciendo constantemente al fluido de tratamiento con el almidón degradado o utilizando un eliminador de oxígeno conjuntamente con dicho almidón degradado. A temperaturas por encima de 300°F, inclusive mediante el uso de las medidas antes mencionadas, los almidones degradados no pueden ser aditivos efectivos para el control de pérdida de fluido.

Se utilizan también polímeros sintéticos lineales convencionales, pero algunas veces requieren otros aditivos, tal como una arcilla, para poner funcionar efectivamente como aditivos destinados a controlar la pérdida de fluido. Sin embargo, el uso de la arcilla puede ser problemático en fluidos de perforación, ya que

la remoción de la arcilla a partir de la formación subterránea puede ser difícil debido a que se infiltra en los poros de la formación subterránea. Además, la adición de arcilla a un fluido de tratamiento aumenta de manera notable la viscosidad del fluido, lo cual puede dar lugar a que se termine la perforación a una velocidad reducida. Otro problema es que algunos polímeros sintéticos no pueden ser utilizados satisfactoriamente de manera conjunta con algunas salmueras, por ejemplo, salmuera divalente. Se cree que esta incompatibilidad evita que el polímero sintético se asocie con la arcilla para formar coloides de enlace, los cuales son deseables debido a que ofrecen cierto grado de pérdida de fluido, por ejemplo, un fluido de perforación.

RESUMEN

La presente invención se refiere a métodos para utilizar aditivos destinados a controlar la pérdida de fluido. En forma más específica, por lo menos en algunas formas, la presente invención se refiere al uso de aditivos para controlar la pérdida de fluido en fluidos de perforación y servicio que comprenden microgeles poliméricos, en operaciones subterráneas.

En una forma, la presente invención proporciona un lodo de perforación que contiene un fluido base acuoso y

un aditivo para el control de pérdida de fluido que contiene por lo menos un microgel polimérico.

En otra forma, la presente invención proporciona un método que comprende suministrar un fluido de tratamiento con base acuosa que contiene un aditivo para controlar la pérdida de fluido y que lleva por lo menos un microgel polimérico; colocar al fluido de tratamiento con base acuosa en una formación subterránea a través de un orificio de pozo que penetra a esta última; permitir que el aditivo para el control de pérdida de fluido quede incorporado en una torta de filtro situada en una superficie dentro de la formación subterránea; permitir que se degrade la torta de filtro; y producir hidrocarburos desde la formación.

En otra forma, la presente invención proporciona un método que comprende suministrar un lodo de perforación que lleva un fluido con base acuosa y un aditivo para el control de pérdida de fluido, el cual comprende por lo menos un microgel polimérico; hacer circular al lodo de perforación en una formación subterránea; permitir que el aditivo para controlar la pérdida de fluido quede incorporado en una torta de filtro situada en una superficie dentro de la formación subterránea; permitir que la torta de filtro sea degradada; y producir hidrocarburos a partir de la formación.

En otra forma, la presente invención proporciona un método que comprende: suministrar una pastilla de tamiz interno que comprende un fluido base acuoso y un aditivo para controlar la pérdida de fluido que lleva por lo menos un microgel polimérico; y colocar a la pastilla de tamiz interno en una formación subterránea, de modo que la misma quede situada entre un tamiz de relleno de grava y un área de la formación subterránea.

Las características y ventajas de la presente invención serán fácilmente evidentes para los expertos en el arte. Si bien los expertos pueden realizar numerosos cambios, los mismos se encuentran dentro del espíritu de la invención.

BREVE DESCRIPCION DE LOS DIBUJOS

Estos dibujos ilustran ciertos aspectos de algunas de las formas de la presente invención y no deben ser utilizados para limitar o definir a la misma.

La figura 1 es una fotografía de una solución transparente de una dispersión de microgel después de disolución en agua.

La figura 2 es una micrografía de la dispersión de microgel de la figura 1.

DESCRIPCION DE LAS FORMAS PREFERIDAS

La presente invención se refiere a métodos para utilizar aditivos destinados al control de pérdida de fluido. Más específicamente, por lo menos en algunas

formas, la presente invención se refiere al uso de aditivos para controlar la pérdida de fluido en fluidos de perforación y servicio que comprenden microgeles poliméricos en operaciones subterráneas.

Pueden existir ventajas potenciales para los métodos y composiciones de la presente invención; se hace alusión aquí a solamente algunos de ellos. La presente solicitud proporciona lodos de perforación, preferiblemente para uso como un lodo o pastillas de perforación o terminación, los cuales forman rápidamente una torta de filtro delgada y degradable. Una vez más, para propósitos de claridad, el término "lodo de perforación" tal como se emplea aquí se refiere generalmente tanto a lodos de perforación como a fluidos de perforación, a menos que se especifique de otro modo. Los fluidos de perforación de la presente invención son menos invasivos para la formación pero sin embargo proporcionan lubricidad, control efectivo de la pérdida de fluido y buenas características de sellado y enlucido de la torta de filtro. La torta de filtro resultante puede ser fácilmente levantada y fácilmente removida o disuelta para ayudar a garantizar poco daño superficial y residual. Sin embargo, los fluidos son estables durante períodos prolongados a alta temperatura. En particular, se cree que los fluidos de perforación de la presente invención tienen rendimiento mejorado en comparación a

los fluidos convencionales que contienen derivados de almidón, por ejemplo, debido a que pueden ser utilizados a temperaturas estáticas efectivas de fondo de pozo hasta de por lo menos 400°F o más, mientras mantienen un efectivo control de la pérdida de fluido y reducen así daños en la formación. Al hacerlo así, está abierta una amplia serie de aplicaciones para el avance tecnológico e ingresos por concepto de producto. Se cree que a concentraciones iguales, los fluidos de perforación de la presente invención que contienen aditivos para el control de pérdida de fluido con los microgeles descritos aquí, dan un rendimiento aumentado (por ejemplo, un orden de incremento de magnitud) en la cantidad de pérdida de fluido, en comparación con materiales de pérdida de fluido basados en almidón.

Otra ventaja potencial puede ser que los fluidos de perforación de la presente invención tienen la capacidad de ser utilizados sin la adición de arcilla, lo cual permite una limpieza más fácil, daños mínimos en la formación, tiempos de perforación más rápidos y la eliminación de los problemas asociados con la viscosidad aumentada de los fluidos cuando se agrega arcilla.

En algunas formas, la presente invención proporciona un lodo de perforación con base acuosa que comprende un aditivo para el control de pérdida de fluido de la presente invención, el cual contiene por lo menos un

microgel polimérico. En otras formas, la presente invención proporciona fluidos de perforación que contienen un fluido base acuoso (por ejemplo, salmuera o agua potable) y uno o más aditivos para controlar la pérdida de fluido que contienen por lo menos un microgel polimérico. En uso práctico, con respecto a la construcción de una torta de filtro que se degradará fácilmente, puede ser ventajoso incluir agente de obturación en el lodo de perforación que quedará incorporado en la torta de filtro. Sin tales agentes de obturación, los microgeles poliméricos pueden formar una torta de filtro efectiva pero la misma puede ser difícil de remover. Otros componentes del lodo de perforación pueden comprender agentes para aumentar la densidad, arcillas, polímeros así como otros aditivos comunes a lodos de perforación con base acuosa, incluyendo pero sin limitarse necesariamente a lubricantes, inhibidores de la corrosión, otros inhibidores y agentes para eliminar oxígeno.

En algunas formas, se puede incluir aditivo para el control de pérdida de fluido en el lodo de perforación, en una cantidad de más o menos 5% en peso o menos, en peso del componente activo del microgel polimérico.

El fluido base de los lodos de perforación de la presente invención puede ser cualquier fluido apropiado con base acuosa pero no está limitado necesariamente a

salmueras (o sea, agua que contiene una sal). En algunos casos, pueden ser apropiadas las salmueras que tienen una densidad de por lo menos 9 libras/galón, referidas como "salmueras de alta densidad". Las salmueras pueden contener esencialmente cualesquiera sales apropiadas incluyendo pero sin limitarse necesariamente a sales basadas en metales, tales como calcio, magnesio, sodio, potasio, cesio, zinc, aluminio y litio. Se prefieren las sales de calcio y zinc. Las sales pueden contener esencialmente cualquier anión; los aniones preferidos son aniones menos costosos que incluyen pero no están limitados necesariamente a cloruros, bromuros, formatos, acetatos y nitratos. En general, el fluido base está presente en un lodo de perforación de la presente invención en una cantidad en la escala desde más o menos 35% hasta más o menos 97% en peso del mismo, de manera más preferible desde aproximadamente 70% hasta más o menos 95% o más.

Preferiblemente, cuando se utilizan, los agentes de obturación son ya sea auto-degradables o degradables en una solución apropiada de limpieza (por ejemplo, un solvente recíproco, agua, una solución ácida, etc.). Ejemplos de agente de obturación apropiados para uso en los métodos de la presente invención incluyen pero no están necesariamente limitados a citrato de magnesio, citrato de calcio, succinato de calcio, maleato de

calcio, tartrato de calcio, tartrato de magnesio, citrato de bismuto, carbonato de calcio, cloruro de sodio y otras sales y sus hidratos. Ejemplos de agentes degradables de enlace pueden incluir pero no están limitados necesariamente a agente de obturación que contienen materiales degradables tales como polímeros degradables. Se considera aquí que un polímero es "degradable" si tal degradación se debe, entre otras cosas, a proceso químico o de radicales tales como hidrólisis, oxidación, degradación enzimática o radiación ultravioleta. Ejemplos apropiados de polímeros degradables que pueden ser empleados de acuerdo con la presente invención incluyen pero no están limitados necesariamente a aquellos descritos en la publicación de Advances in Polymer Science, Vol. 157, titulada "Degradable Aliphatic Polyesters" editado por A C. Albertson, cuya descripción se incorpora aquí a modo de referencia. Ejemplos específicos de polímeros apropiados incluyen pero no están limitados necesariamente a polisacáridos tales como dextranos o celulosas; quitinas; quitosanos; proteínas; orto-ésteres; poliésteres alifáticos; poli(láctidos); poli(glicólicos); poli(hidroxi butiratos); poli(anhídridos); policarbonatos alifáticos; poli(ortoésteres); ácidos (poliamino); óxidos de polietileno) y polifosfazenos. También son apropiados las combinaciones y derivados de los mismos.

Cuando se elige un agente particular de obturación, se debe conocer el rendimiento del mismo a la escala de temperatura de la aplicación. Los agentes de obturación utilizados pueden estar presentes generalmente en las composiciones de lodo de perforación en una cantidad en la escala desde más o menos 1% hasta más o menos 40% en peso del mismo, de manera más preferible desde más o menos 5% hasta más o menos 25%. En general, los agentes de obturación pueden tener un tamaño de partícula en la escala desde más o menos 1 micra hasta más o menos 600 micras. Preferiblemente, el tamaño de la partícula de obturación está en la escala desde más o menos 1 hasta más o menos 200 micras, pero puede variar de formación a formación. El tamaño de partícula utilizado está determinado por el tamaño de la garganta de los poros de la formación.

El término "microgel polimérico", tal como se emplea aquí, se refiere a una partícula en forma de gel que contiene una red de polímero degradado (por ejemplo, soluble o dilatable en agua). En algunas formas, el aditivo para el control de pérdida de fluido puede ser suministrado en un medio continuo, por ejemplo, en una forma de dispersión. En otras formas, los microgeles poliméricos pueden ser utilizados sin ningún medio continuo, por ejemplo, en forma seca. Una de las muchas características útiles de ciertas formas de los

microgeles poliméricos de la presente invención es que se cree que dilatan o encogen en respuesta a estímulos externos, por ejemplo, pH y temperatura. En algunas formas, algunos de los microgeles poliméricos pueden ser considerados como super-absorbentes puesto que contienen más de 99% de agua. Esto puede ser ventajoso en algunas aplicaciones en las cuales es importante la absorción de agua.

Cuando hay una porción importante del aditivo para el control de pérdida de fluido en un lodo de perforación de la presente invención, los microgeles poliméricos pueden ser dispersados en un medio continuo antes de la adición al fluido. En algunas formas, el aditivo para el control de pérdida de fluido puede comprender un medio continuo. Ejemplos de medios continuos apropiados pueden incluir pero no están limitados necesariamente a fluidos con base acuosa, alcoholes, glicerina, glicoles, poliglicol aminas, polioles y cualquier derivado de los mismos. Además, en algunas formas, el medio continuo puede contener un fluido seleccionado a partir del grupo que consiste de metanol, etanol, n-propanol, isopropanol, n-butanol, butanol secundario, isobutanol, t-butanol, una mezcla de metanol, etanol, n-propanol, isopropanol, n-butanol, butanol secundario, isobutanol o t-butanol y agua; una mezcla de sulfato de amonio, sulfato de sodio o sulfato de potasio y agua, una mezcla de cloruro de

sodio, cloruro de potasio o cloruro de calcio y agua; y sus combinaciones. Opcionalmente, el medio continuo comprende un fluido seleccionado a partir del grupo que consiste de etanol, una mezcla de t-butanol y agua; y una mezcla de sulfato de amonio y agua. También son apropiadas las mezclas de tales compuestos. Ejemplos de fluidos apropiados con base acuosa pueden incluir pero no están necesariamente limitados a agua dulce, agua de mar y salmueras (por ejemplo, aguas saladas saturadas). Ejemplos de salmueras apropiadas incluyen pero no están limitadas necesariamente a salmueras monovalentes, salmueras divalentes y salmueras trivalentes que contienen sales solubles tales como cloruro de sodio, cloruro de calcio, bromuro de calcio, bromuro de zinc, carbonato de potasio, formato de sodio, formato de potasio, formato de cesio, acetato de sodio, acetato de potasio, acetato de calcio, acetato de amonio, cloruro de amonio, bromuro de amonio, nitrato de sodio, nitrato de potasio, citrato de amonio, sulfato de amonio, nitrato de calcio, carbonato de sodio, carbonato de potasio, cualquier combinación de los mismos y cualquier derivado de ellos. Ejemplos de alcoholes apropiados incluyen pero no están limitados necesariamente a metanol, etanol, propanol, isopropanol, butanol, butanol terciario, etc. Ejemplos de glicoles apropiados incluyen pero no están limitados necesariamente a poliglicoles, propileno

glicol, etileno glicol, etc. En algunas formas, el medio continuo puede estar presente en cualquier escala apropiada que incluye proporciones de medio continuo a microgel polimérico de más o menos 30:70, más o menos 20:80, más o menos 10:90, más o menos 5:95 y más o menos 0:100 por ciento en peso.

En algunas formas, los microgeles poliméricos pueden comprender un producto de reacción formado por medio de una reacción apropiada de polimerización de un polímero (o monómero) y un agente de degradación. En una forma de un método para formar microgeles de la presente invención, la polimerización es una polimerización por dispersión en un medio continuo que es esencialmente inerte a las reacciones de transferencia de cadena. Se forman partículas de microgel de polímero degradado que se cree son insolubles o cuando más dilatables en el medio continuo. Si bien no se desea limitarse a ninguna teoría en particular, se cree que en algunas formas el agente de degradación puede actuar como un iniciador que forma ramificaciones de cadena del polímero o monómero, las cuales pueden reaccionar a su vez entre si para formar un microgel polimérico. Otros iniciadores apropiados pueden incluir aquellos conocidos por los expertos en el arte, tales como foto-iniciadores, iniciadores térmicos y sus combinaciones.

En algunas formas, el polímero (o monómero) puede comprender cualquier polímero o monómero apropiado que pueda formar un microgel polimérico cuando se degrada. Los polímeros y monómeros apropiados pueden incluir pero no están limitados necesariamente a aquellos que comprenden unidades basadas en acrilamidas, alcoholes vinílicos, vinil pirrolidonas, vinilpiridinas, acrilatos, poliacrilamidas, alcoholes polivinílicos, polivinil pirrolidonas, polivinil piridinas, acrilatos, poliacrilamidas, alcoholes polivinílicos, polivinil pirrolidonas, polivinil piridinas, poliacrilatos, polibuteno succinato, polibuteno succinato-coadipato, polihidroxi butirato-valerato, polihidroxi butirato-covalerato, policaprolactonas, amidas poliéster, tereftalatos de polietileno, tereftalato de polietileno sulfonatado, óxidos polietileno, polietilenos, polipropilenos, copoliéster aromáticos alifático, ácidos poliacrílicos, polisacáridos (tales como dextran o celulosa), quitinas, quitosanos, proteínas, poliésteres alifáticos, ácidos polilácticos, poli(glicolidos), poli(ϵ -caprolactonas), éteres poli(hidroxi éster), poli(hidroxi butiratos), poli(anhídridos), policarbonatos, poli(orto ésteres), ácidos (poliamino), óxidos poli(etileno) , óxidos (polipropileno), poli(fosfazenos), amidas poliéster, poliamidas, poli-estirenos, cualquier derivado de los mismos, cualquier

copolímero, homopolímero o terpolímero o cualquier mezcla de ellos. En algunas formas, el monómero puede incluir un grupo no saturado, tal como un monómero que incluye un grupo vinilo. Ejemplos de monómeros que contienen vinilo pueden ser descritos mediante la fórmula $C(R_1)(R_2)=C(R_3)(R_4)$, en la cual R_1 , R_2 , R_3 y R_4 son segmentos que producen solubilidad o dilatación de este monómero en el solvente común. Opcionalmente, R_1 , R_2 , R_3 y R_4 pueden estar seleccionados cada uno en forma independiente a partir de pero sin limitarse a hidrógeno, metilo, etilo, $CONH_2$, $CONHCH_3$, $CON(CH_3)_2$, CH_2SO_3H , CH_2SO_3Na y $COONa$. En una opción adicional, el monómero comprende un compuesto seleccionado a partir del grupo que consiste de hidroxietil acrilato, acrilamida e hidroxietil metacrilato. Ejemplos de copolímeros apropiados que pueden ser utilizados con los mismos pueden incluir polímeros inorgánicos y orgánicos. Algunos de estos polímeros y monómeros pueden ser más apropiados para aplicaciones con una temperatura menos alta que otras; sin embargo, la degradación puede aumentar su estabilidad térmica. En algunas formas, el polímero o monómero puede comprender un polímero o monómero soluble en agua. En algunas formas, el polímero o monómero puede comprender un copolímero de bloque con una porción que es soluble en agua y una porción que es insoluble en agua.

En algunas formas, el agente de degradación puede comprender cualquier agente que pueda reaccionar con un polímero o monómero para formar un microgel polimérico. En algunas formas, el agente de degradación puede comprender un agente covalente de degradación. Ejemplos de agentes apropiados de degradación pueden incluir agentes que forman un radical. Los agentes apropiados de degradación pueden incluir pero no están limitados a 2,2'-azobis-(2-metil butironitrilo), 2,2'-azobis(isobutiramida hidrocloreto), 2,2'-azobis[2-imidazolin-2-il]propano]dihidrocloreto, 1,1'-azobis(ciclohexanol carbonitrilo), 2,2'-azobis(2-metil propionamida dihidrocloreto, ácido 4,4'-azobis(4-cianovalérico), persulfato de amonio, dihidrato de la sal monosódica de ácido hidroximetanosulfónico, persulfato de potasio, persulfato de sodio, peróxido de benzoilo, 1,1-bis(amil peroxi terciario)ciclohexano, 1,1-bis(butilperoxi terciario) ciclohexano, 2,2-bis(butilperoxi terciario)ciclohexano, 2,2-bis(butilperoxi terciario)butano, peróxido de 2,4-pentanodiona, 2,5-bis(butilperoxi terciario)-2,5-dimetilhexano, 2,5-di(butilperoxi terciario)-2,5-dimetil-3-hexino; peróxido de 2-butanona, hidroperóxido de cumeno, di-terciario-amil peróxido, dicumil peróxido, lauroil peróxido, hidroperóxido butílico terciario, peracetato de butilo terciario, peróxido de butilo terciario,

peroxibenzoato de butilo terciario, butilperoxi terciario-2-etilhexil carbonato, dietileno glicol dimetacrilato, azobis-isobutironitrilo y sus combinaciones. Un experto en el arte podría seleccionar un agente apropiado de degradación basado en el polímero o monómero a ser degradado.

En algunas formas, la cantidad del agente de degradación utilizado conjuntamente con el polímero o monómero puede ser cualquier cantidad necesaria para lograr el grado deseado de degradación y otras características. En algunas formas, la relación de agente de degradación a monómero o polímero utilizado para formar al microgel polimérico puede estar en la escala desde más o menos 100:1 hasta más o menos 5.000:1 en peso.

En una forma de un método para formar a los microgeles de la presente invención, la polimerización es una polimerización por dispersión en un medio continuo tal como un medio continuo que contiene alcohol t-butílico/agua y sulfato de amonio. Tanto el monómero (tal como pero sin limitarse a acrilamida) y el agente de degradación (tal como pero sin limitarse a N,N'-etileno-bis-acrilamida) son esencialmente solubles o por lo menos dilatables en el medio continuo. Los microgeles resultantes no son solubles en el medio continuo. Se cree que este medio continuo es relativamente inerte (o

por lo menos no es tan reactivo para alterar la formación de los microgeles) a las reacciones de transferencia de cadena mediante la propagación de radicales. Mediante la extracción del medio continuo utilizando una técnica apropiada, si se desea se puede secar a los microgeles.

En forma opcional, se puede incluir también un estabilizador coloidal en el medio continuo durante la polimerización. Un ejemplo de un estabilizador coloidal es anfifílico, tal como un estabilizador coloidal anfifílico que contiene un estabilizador seleccionado a partir del grupo que consiste de poli(vinil pirrolidona) (PVP), cloruro de polidialil dimetil amonio (poli-DADMAC) y sus combinaciones.

En una forma de un método para formar a los microgeles de la presente invención, la polimerización es una polimerización por dispersión de monómeros vinílicos solubles o dilatables en agua (representados por $C(R_1)(R_2)=C(R_3)(R_4)$, en la cual R_1 , R_2 , R_3 y R_4 son segmentos que producen la solubilidad o dilatabilidad de este monómero en agua y del medio de polimerización en presencia de un agente de degradación radicalmente polimerizable (tal como pero sin limitarse a $C(R_5)(R_6)=C(R_7)-(R_8)-C(R_9)=C(R_{10})(R_{11})$, donde R_5-R_{11} son grupos que permiten la solubilidad o capacidad de dilatación de este agente de degradación en agua y del

medio de polimerización) en un medio (tal como pero sin limitarse a t-butanol/agua y solución de sulfato de amonio en agua) para el cual se evita o suprime la transferencia de cadena del radical de propagación. Aprovechando la estabilidad deficiente de la dispersión resultante a un alto contenido de polímero (por ejemplo, 13% en peso), en ausencia de cualquier agente de degradación, el resultado puede ser un pedazo de macrogel.

La escala de tamaño para estos microgeles poliméricos puede variar desde más o menos 10 mm hasta más o menos 1 mm de diámetro. Una escala apropiada de tamaño puede ser 10 micras hasta más o menos 500 micras. En una determinada composición se puede utilizar una escala de microgeles poliméricos de tamaño desigual. Un experto en el arte podría seleccionar un tamaño apropiado de los microgeles poliméricos, tomando como base las características de la formación subterránea en la cual se podría utilizar a tales microgeles, la temperatura de la formación subterránea, el grado de estabilidad requerido de los microgeles poliméricos y los costos. Del mismo modo, pueden variar las formas de los microgeles poliméricos y pueden depender de varios factores que incluyen pero no están limitados necesariamente a temperatura, medio continuo utilizado y grado de degradación. Los microgeles poliméricos pueden

tener cualquier forma, por ejemplo, por lo menos parcialmente esférica. Dependiendo del método en el cual se forman los microgeles poliméricos, puede variar la forma de los mismos. Un experto en el arte podría seleccionar una forma apropiada de los microgeles poliméricos, tomando como base las características de la formación subterránea en la cual se podría utilizar a los mismos, la temperatura de esta última, el grado de estabilidad requerido de los microgeles poliméricos y los costos. Ninguna forma particular es crítica para uso en la presente invención.

En algunas formas, los aditivos para el control de fluido de la presente invención no pueden ser suministrados con un medio continuo. Estas pueden ser referidas como las formas "secas". Por ejemplo, en algunas formas, los microgeles poliméricos son polimerizados, los microgeles poliméricos pueden ser extraídos a partir del medio continuo y luego secados utilizando una técnica apropiada para formar a los microgeles poliméricos en polvo.

En algunas formas, la presente invención comprende un fluido de perforación con base acuosa que lleva un aditivo para controlar la pérdida de fluido, de la presente invención, que comprende por lo menos un microgel polimérico. En algunas formas, el aditivo para el control de la pérdida de fluido puede ser incluido en

el fluido de perforación en una cantidad de más o menos 5% o menos en peso del componente activo del microgel polimérico. Otros componentes de los fluidos de perforación pueden comprender agentes de obturación y otros aditivos comúnmente utilizados.

En algunas formas, la presente invención comprende una pastilla de tamiz interno que contiene un fluido basado en agua y un aditivo para el control de pérdida de fluido, el cual contiene por lo menos un microgel polimérico. En algunas formas, el aditivo para el control de la pérdida de fluido puede ser incorporado en la pastilla de tamiz interno en una cantidad de más o menos 1 a más o menos 10% en peso del compuesto activo del microgel polimérico.

En algunas formas, la presente invención proporciona un lodo de perforación que contiene un fluido con base acuosa y un aditivo para el control de la pérdida de fluido que contiene por lo menos un microgel polimérico.

En algunas formas, la presente invención proporciona un método que comprende proporcionar un fluido de tratamiento con base acuosa que contiene un aditivo para controlar la pérdida de fluido, el cual contiene un microgel polimérico; y colocar al fluido de tratamiento con base acuosa en una formación subterránea a través de un orificio de pozo que penetra a la misma.

En algunas formas, la presente invención proporciona un método que comprende: proporcionar un lodo de perforación que contiene un fluido con base acuosa y un aditivo para controlar la pérdida de fluido que contiene por lo menos un microgel polimérico; y hacer circular al lodo de perforación en una formación subterránea.

En algunas formas, la presente invención proporciona un método que comprende: proporcionar una pastilla de tamiz interno que contiene un fluido con base acuosa y un aditivo para controlar la pérdida de fluido que contiene por lo menos un microgel polimérico; y colocar a la pastilla de tamiz interno en una formación subterránea, de modo que tal pastilla quede situada entre un tamiz de relleno de grava y un área de la formación subterránea.

Para facilitar una mejor comprensión de la presente invención, se ofrece los siguientes ejemplos de ciertos aspectos de algunas formas. De ningún modo se debe interpretar que los siguientes ejemplos limitan o definen el alcance completo de la invención .

EJEMPLOS

Ejemplo 1

Se preparó partículas de microgel polimérico de acuerdo con el siguiente procedimiento. Se preparó una mezcla de 95,0 gramos de etanol, 20,0 gramos de acrilamida, 0,50 gramos de polivinil pirrolidona y 0,50

gramos de dietileno glicol dimetacrilato, en un matraz de tres cuellos y capacidad de 250 mL, equipado con un condensador y un agitador mecánico. Se purgó a la mezcla con gas nitrógeno durante 30 minutos y luego se calentó hasta 65°C. Luego, se inyectó en la mezcla 5,0 gramos de una solución de azobis isobutironitrilo en etanol (1% de azobis isobutironitrilo en peso). Se mantuvo a la mezcla a 65°C bajo nitrógeno y se agitó durante 22 horas a 300 rpm. Luego, se dejó enfriar a la mezcla hasta temperatura ambiente y se obtuvo una dispersión de partículas con tamaños desde 10 μm hasta 100 μm (en agua) con un contenido de sólidos de aproximadamente 17% en peso. La figura 1 es una fotografía de una dispersión de microgel transparente formada en este ejemplo. La figura 2 es una micrografía de la dispersión de microgel formada en este ejemplo. Luego, las dispersiones fueron centrifugadas a 4800 rpm durante 5 minutos. Después de esto, se decantó al líquido sobrenadante y se recogió los sedimentos, los cuales fueron derivados al vacío a aproximadamente 50°C durante toda la noche para producir polvos secos sueltos bien dispersados.

Ejemplo 2

Se preparó partículas de microgel polimérico de acuerdo con el siguiente procedimiento. Se preparó una mezcla de 95,0 gramos de una mezcla de t-butanol y agua (90% de t-butanol en peso), 15,0 gramos de acrilamida,

0,20 gramos de N,N'-etileno-bis-acrilamida y 0,50 gramos de poli(vinil pirrolidona), en un matraz con tres cuellos, capacidad de 250 mL, equipado con un condensador y un agitador mecánico. Se purgó a la mezcla con gas nitrógeno durante 30 minutos y luego se calentó hasta 65°C. Luego, se inyectó en la mezcla 5,0 gramos de solución de azobis isobutironitrilo en etanol (1% de azobis isobutironitrilo en peso). Después de eso, se dejó enfriar a la mezcla hasta temperatura ambiente y se obtuvo una dispersión de partículas con tamaños desde 10 µm hasta 300 µm, con un contenido de sólidos de aproximadamente 13% en peso.

Ejemplo 3

Se preparó partículas de microgel polimérico de acuerdo con el siguiente procedimiento. Se preparó una mezcla de 75,5 gramos de una solución al 40% de sulfato de amonio en agua, 7,5 gramos de acrilamida, 12,0 gramos de una solución al 20% de cloruro de polidialil dimetil amonio en agua, 6,7 µL de poli (etileno glicol) diacrilato y 20,0 gramos de agua desionizada en un matraz con tres cuellos y capacidad de 250 mL, equipado con un condensador y un agitador mecánico. Se purgó a la mezcla con gas nitrógeno durante 30 minutos y luego se calentó hasta 35°C. Después se inyectó en la mezcla una solución de 5,0 gramos de 2,2'-azobis[2-(2-imidazolin-2-il)propano]dihidrocloruro en agua (0,01% de

2,2'-azobis[2-(2-imidazolin-2-il)propano]dihidrocloruro en peso). Luego, se mantuvo a la mezcla a 35°C bajo nitrógeno y se agitó durante 22 horas a 300 rpm. Después de eso, se dejó enfriar a la mezcla hasta temperatura ambiente y se obtuvo una dispersión de partículas con un contenido de sólidos de aproximadamente 9% en peso. Luego, las dispersiones fueron centrifugadas a 4800 rpm durante 5 minutos. Después, se derivó a los sedimentos al vacío a aproximadamente 50°C durante toda la noche para producir polvos secos sueltos y bien dispersados.

Ejemplo 4

Se preparó partículas de microgel polimérico de acuerdo con el siguiente procedimiento. Se preparó una mezcla de t-butanol y agua (90% de t-butanol en peso), 15,0 gramos de acrilamida, 0,50 gramos de poli(vinil pirrolidona), en un matraz de tres cuellos con capacidad de 250 mL, equipado con un condensador y un agitador mecánico. Se purgó a la mezcla con gas nitrógeno durante 30 minutos y luego se calentó hasta 65°C. Luego, se inyectó una solución de 5,0 gramos de azobis isobutironitrilo en etanol (1% de azobis isobutironitrilo en peso) en la mezcla. Después de aproximadamente 20 minutos, se inyectó en la mezcla una solución de 0,10 gramos de N,N'-etileno-bis.-acrilamida en 5,0 gramos de una mezcla de t-butanol y agua (90% de t-butanol en peso) . Luego, se mantuvo a la mezcla a

65°C bajo nitrógeno y se agitó durante 22 horas a 300 rpm. Después, se dejó enfriar a la mezcla hasta temperatura ambiente y se obtuvo una dispersión de partículas con tamaños desde 10 μm hasta 300 μm con un contenido de sólidos de aproximadamente 13% en peso. Luego, se centrifugó a las dispersiones a 4800 rpm durante 5 minutos. Luego, se decantó al líquido sobrenadante y se recogió los sedimentos, los cuales fueron secados al vacío a aproximadamente 50°C durante toda la noche para producir polvos secos sueltos y bien dispersados.

Ejemplo 5

Se preparó partículas de microgel polimérico de acuerdo con el siguiente procedimiento. Se preparó una mezcla de 75,5 gramos de una solución al 40% de sulfato de amonio en agua, 7,5 gramos de acrilamida, 12,0 gramos de solución al 20% de cloruro de polidialil dimetilamonio en agua, 67 μL de poli(etileno glicol) diacrilato y 20,0 gramos de agua desionizada, en un matraz con tres cuellos y capacidad de 250 mL, equipado con un condensador y un agitador mecánico. Se purgó a la mezcla con gas nitrógeno durante 30 minutos y luego se calentó hasta 35°C. Luego, se inyectó en la mezcla una solución de 5,0 gramos de 2,2'-azobis{2(2-imidazolin-2-il)propano} dihidrocloruro en agua (0,01% de 2,2'-azobis[2-(2-imidazolin-2-il)propano]dihidrocloruro en

peso). Luego, se mantuvo a la mezcla a 35°C bajo nitrógeno y se agitó durante 22 horas a 300 rpm. Después, se dejó enfriar a la mezcla hasta temperatura ambiente y se obtuvo una dispersión de partículas con un contenido de sólidos de aproximadamente 9% en peso. Luego, se centrifugó a las dispersiones a 4800 rpm durante 5 minutos. Luego, se decantó al líquido sobrenadante y se recogió los sedimentos, los cuales fueron derivados al vacío a aproximadamente 50°C durante toda la noche para producir polvos secos sueltos y bien dispersados.

Ejemplo 6

Se preparó la composición de prueba de acuerdo con el siguiente procedimiento. Se preparó una mezcla de 75,5 gramos de una solución al 40% de sulfato de amonio en agua, 7,5 gramos de acrilamida, 12,0 gramos de una solución al 20% de cloruro de polidialil dimetilamonio en agua y 20,0 gramos de agua desionizada, en un matraz con tres cuellos y capacidad de 250 mL, equipado con un condensador y un agitador mecánico. Se purgó a la mezcla con gas nitrógeno durante 30 minutos y luego se calentó hasta 35°C. Luego, se inyectó en la mezcla 5,0 gramos de una solución de 2,2'-azobis[2(2-imidazolin-2-il)propano] dihidrocloruro en agua (0,01% de 2,2'-azobis[2-(2-imidazolin-2-il)propano]dihidrocloruro en peso). Luego, se mantuvo a la mezcla a 35°C bajo

nitrógeno y se agitó durante 22 horas a 300 rpm. Después, se dejó enfriar a la mezcla hasta temperatura ambiente y se obtuvo una dispersión de partículas con un contenido de sólidos de aproximadamente 9% en peso.

Ejemplo 7

Se preparó la composición de prueba 2 de acuerdo con el siguiente procedimiento. Se preparó una mezcla de 75,5 gramos de una solución al 40% de sulfato de amonio en agua, 7,5 gramos de acrilamida, 12,0 gramos de una solución al 20% de cloruro de polidialil dimetilamonio en agua, 6,7 µL de poli(etileno glicol)diacrilato y 20,0 gramos de agua desionizada, en un matraz con tres cuellos y capacidad de 250 mL, equipado con un condensador y un agitador mecánico. Se purgó a la mezcla con gas nitrógeno durante 30 minutos y luego se calentó hasta 35°C. Luego, se inyectó en la mezcla 5,0 gramos de una solución de 2,2'-azobis[2(2-imidazolin-2-il)propano]dihidrocloruro en agua (0,01% de 2,2'-azobis[2-(2-imidazolin-2-il)propano]dihidrocloruro en peso). Luego, se mantuvo a la mezcla a 35°C bajo nitrógeno y se agitó durante 22 horas a 300 rpm. Después, se dejó enfriar a la mezcla hasta temperatura ambiente y se obtuvo una dispersión de partículas con un contenido de sólidos de aproximadamente 9% en peso.

Ejemplo 8

Se preparó la composición de prueba 3 de acuerdo con el siguiente procedimiento. Se preparó una mezcla de 90,0 gramos de una mezcla de t-butanol y agua (90% en peso de t-butanol), 15,0 gramos de acrilamida, 0,50 gramos de poli(vinil pirrolidona), en un matraz con tres cuellos y capacidad de 250 mL, equipado con un condensador y un agitador mecánico. Se purgó a la mezcla con gas nitrógeno durante 30 minutos y luego se calentó hasta 65°C. Luego, se inyectó en la mezcla 5,0 gramos de una solución de azobis isobutironitrilo en etanol (1% en peso de azobis isobutironitrilo). Después de aproximadamente 20 minutos, se inyectó en la mezcla una solución de 0,10 gramos de N,N'-etileno-bis-acrilamida en 5,0 gramos de una mezcla de t-butanol y agua (90% en peso de t-butanol). Luego, se mantuvo a la mezcla a 65°C bajo nitrógeno y se agitó durante 22 horas a 300 rpm. Después, se dejó enfriar a la mezcla hasta temperatura ambiente y se obtuvo una dispersión de partículas con tamaños desde 10 μm hasta 300 μm , con un contenido de sólidos de aproximadamente 13% en peso. Luego, se centrifugó a las dispersiones a 4800 rpm durante 5 minutos. Después se decantó al líquido sobrenadante y se recogió los sedimentos, los cuales fueron derivados al vacío a aproximadamente 50°C durante toda la noche para producir polvos secos sueltos y bien dispersados.

Ejemplo 9

Se preparó la composición de prueba 4 de acuerdo con el siguiente procedimiento. Se preparó una mezcla de 90,0 gramos de t-butanol y agua (90% en peso de t-butanol), 15,0 gramos de acrilamida, 0,50 gramos de poli(vinil pirrolidona), en un matraz con tres cuellos y capacidad de 250 mL, equipado con un condensador y un agitador mecánico. Se purgó a la mezcla con gas nitrógeno durante 30 minutos y luego se calentó hasta 65°C. Luego, se inyectó en la mezcla 5,0 gramos de una solución de azobis isobutironitrilo en etanol (1% en peso de azobis isobutironitrilo). Después de aproximadamente 20 minutos se inyectó en la mezcla una solución de 0,40 gramos de N,N'-etileno-bis-acrilamida en 5,0 gramos de una mezcla de t-butanol y agua (90% en peso de t-butanol). Luego, se mantuvo a la mezcla a 65°C bajo nitrógeno y se agitó durante 22 horas a 300 rpm. Después, se dejó enfriar a la mezcla hasta temperatura ambiente y se obtuvo una dispersión de partículas con un contenido de sólidos de aproximadamente 13% en peso. Luego, se centrifugó las dispersiones a 4800 rpm durante 5 minutos. Se decantó al líquido sobrenadante y se recogió los sedimentos, los cuales fueron derivados al vacío a aproximadamente 50°C durante toda la noche para producir polvos secos sueltos y bien dispersados.

Ejemplo 10

Se preparó la composición de prueba 5 de acuerdo con el siguiente procedimiento. Se preparó una mezcla de 90,0 gramos de t-butanol y agua (90% en peso de t-butanol), 15,0 gramos de acrilamida, 0,50 gramos de poli(vinil pirrolidona), en un matraz con tres cuellos y capacidad de 250 mL, equipado con un condensador y un agitador mecánico. Se purgó a la mezcla con gas nitrógeno durante 30 minutos y luego se calentó hasta 65°C. Luego, se inyectó en la mezcla 5,0 gramos de una solución de azobis isobutironitrilo en etanol (1% en peso de azobis isobutironitrilo). Después de aproximadamente 20 minutos se inyectó en la mezcla una solución de 0,20 gramos de N,N'-etileno-bis-acrilamida en 5,0 gramos de una mezcla de t-butanol y agua (90% en peso de t-butanol). Luego, se mantuvo a la mezcla a 65°C bajo nitrógeno y se agitó durante 22 horas a 300 rpm. Después, se dejó enfriar a la mezcla hasta temperatura ambiente y se obtuvo una dispersión de partículas con un contenido de sólidos de aproximadamente 13% en peso. Luego, se centrifugó las dispersiones a 4800 rpm durante 5 minutos. Se decantó al líquido sobrenadante y se recogió los sedimentos, los cuales fueron derivados al vacío a aproximadamente 50°C durante toda la noche para producir polvos secos sueltos y bien dispersados.

Ejemplo 11

Se preparó la composición de prueba 6 de acuerdo con el siguiente procedimiento. Se preparó una mezcla de 90,0 gramos de t-butanol y agua (90% en peso de t-butanol), 15,0 gramos de acrilamida, 0,50 gramos de poli(vinil pirrolidona), en un matraz con tres cuellos y capacidad de 250 mL, equipado con un condensador y un agitador mecánico. Se purgó a la mezcla con gas nitrógeno durante 30 minutos y luego se calentó hasta 65°C. Luego, se inyectó en la mezcla 5,0 gramos de una solución de azobis isobutironitrilo en etanol (1% en peso de azobis isobutironitrilo). Después de aproximadamente 20 minutos se inyectó en la mezcla una solución de 0,050 gramos de N,N'-etileno-bis-acrilamida en 5,0 gramos de una mezcla de t-butanol y agua (90% en peso de t-butanol). Luego, se mantuvo a la mezcla a 65°C bajo nitrógeno y se agitó durante 22 horas a 300 rpm. Después, se dejó enfriar a la mezcla hasta temperatura ambiente y se obtuvo una dispersión de partículas con un contenido de sólidos de aproximadamente 13% en peso. Luego, se centrifugó las dispersiones a 4800 rpm durante 5 minutos. Se decantó al líquido sobrenadante y se recogió los sedimentos, los cuales fueron derivados al vacío a aproximadamente 50°C durante toda la noche para producir polvos secos sueltos y bien dispersados.

Ejemplo 12

Se preparó la composición de prueba 7 de acuerdo con el siguiente procedimiento. Se mezcló cantidades iguales de los polvos procedentes de los Ejemplos 8, 9, 10 y 11 para producir un aditivo en polvo para el control de la pérdida de fluido.

Ejemplo 13

Se realizó pruebas de pérdida de fluido sobre las composiciones 1-7 de acuerdo con el siguiente procedimiento. Se agregó dosis variables de cada una de las composiciones de prueba, así como del aditivo convencional para controlar la pérdida de fluido N-Drill HT Plus® (disponible comercialmente en Halliburton Energy Services, Inc., Houston, Texas) para separar composiciones de lodo de perforación que contienen 0,09 gramos de agente para impartir viscosidad N-VIS® (comercialmente disponible en Halliburton Energy Services, Inc., Houston, Texas), 14,0 gramos de agente de obturación BARACARB® 5 (comercialmente disponible en Halliburton Energy Services, Inc., Houston, Texas), 3,5 gramos de agente de obturación CARACARB® 5 Halliburton Energy Services, Inc., Houston, Texas) y 137 gramos de una solución al 26% en peso de NaCl en agua. Se redujo la cantidad de NaCl en los lodos de perforación, que agregan los aditivos de control de pérdida de fluido de los Ejemplos 6 y 7, para tomar en cuenta la salmuera presente en tales aditivos. Se colocó a cada uno de los

41

lodos de perforación en una Prensa de Filtro de 175 mL de "OFITE HPHT para evaluar la pérdida de fluido. En un experimento típico, se ajustó las presiones hasta 600 y 100 libras por pulgada cuadrada (700 y 200 libras por pulgada cuadrada cuando la temperatura está por encima de 300°F) en la parte superior e inferior de una cámara de muestra, la cual fue llenada con lodo y precalentada hasta la temperatura de prueba. Se recogió al líquido drenado procedente de un papel filtro con una porosidad de 2,7 μm , sellado en el fondo del cilindro. Se ofrece en la Tabla 1 el rendimiento de cada aditivo para el control de pérdida de fluido. El rendimiento aceptable de un aditivo fue establecido en 10 ML volúmenes de filtrado con una prueba de 30 minutos.

Tabla I

Aditivo Para el Control de Pérdida de Fluido	Temperatura (°F)	Agente de Degradación	Dosis (% en peso)	Pérdida de Fluido (mL)
N-DRILL HT PLUS®	250	N/A	2,0	9,5
Composición de Prueba 1	250	0	2,0	100
Composición de Prueba 2	250	0,006	2,0	4,3
N-DRILL HT PLUS®	300	N/A	2,0	100
Composición de Prueba 2	300	0,006	2,0	100
Composición de Prueba 2	300	0,006	1,0	32
Composición de Prueba 2	300	0,006	0,5	37
Composición de Prueba 3	300	0,7	2,0	6,3
Composición de Prueba 3	300	0,7	1,0	16,3
Composición de Prueba 3	300	0,7	0,5	50,0
Composición de Prueba 3	350	0,7	2,0	40,0
Composición de Prueba 4	350	2,7	2,0	10,2
Composición de Prueba 5	350	1,3	2,0	7,5
Composición de Prueba 6	350	0,3	2,0	43,5
Composición de Prueba 7	350	1,3	2,0	6,5

En algunas formas, la presente invención proporciona un método que comprende: suministrar un fluido de tratamiento con base acuosa que contiene un aditivo para el control de pérdida de fluido que lleva por lo menos un microgel polimérico; colocar al fluido de tratamiento con base acuosa en una formación subterránea a través de un orificio de pozo que penetra a la misma; permitir que el aditivo para el control de pérdida de fluido quede incorporado en una torta de filtro situada sobre una superficie dentro de la formación subterránea; permitir

que se degrade la torta de filtro; y producir hidrocarburos a partir de la formación.

Como se puede apreciar en la Tabla I, la composición de prueba 1 que representaba un polímero de alto peso molecular en calidad de forro no presentó efectividad como casi todo el fluido drenado. La composición de prueba 2 (en solución de salmuera) funcionó mejor que N-Drill HT Plus® (pérdida de fluido de 4,3 mL en comparación a una pérdida de fluido de 9,5 mL) a la misma dosis y a 250°F. Sin embargo, cuando se probó a 300°F, tanto la composición de prueba 2 como N-Drill HT Plus® fallaron casi completamente durante la prueba a una dosis de 2 por ciento en peso. La composición 2 pudo reducir la pérdida de fluido hasta 32 mL y 37 mL a una dosis de 1 por ciento en peso y 0,5 por ciento en peso respectivamente.

A 300°F, la composición de prueba 3 (contenido de agente de degradación de aproximadamente 0,7 por ciento en peso) controló la pérdida de fluido alrededor de 6,3 mL, pero sin embargo este aditivo no pudo satisfacer los criterios de rendimiento aceptable de un volumen de 10 mL del filtrado en 30 minutos a 350°F. Las composiciones de prueba 5 y 7 (contenido de agente de degradación de aproximadamente 1,3 por ciento en peso) pudieron controlar la pérdida de fluido alrededor de 7,5 y 6,5 mL a 350°F respectivamente. Sin embargo, el aumento del

contenido de agente de degradación hasta 2,7 por ciento en peso (según lo demostrado por la composición 4) dio como resultado una pérdida de fluido de 10,2 mL.

Como se puede apreciar en la Tabla 1 y demostrado adicionalmente por la Figura 3, la dosis tiene un efecto sobre el control de la pérdida de fluido. Tal como se ilustra en la Figura 3, la pérdida de fluido mediante la composición de prueba 3 disminuyó desde 50 mL hasta 6,3 mL ya que la dosis de este aditivo aumentó desde 0,5 por ciento en peso hasta 2,0 por ciento en peso. Así, es altamente probable que la pérdida de fluido pudiera ser reducida adicionalmente a dosis superiores a 2,0 por ciento en peso, aún cuando podría existir una dosis ideal.

Como se puede apreciar en la Tabla 1, el volumen del filtrado disminuyó desde 43,5 mL hasta 7,5 mL, cuando el contenido del agente de degradación del correspondiente aditivo de pérdida de fluido aumentó desde 0,3 por ciento en peso hasta 1,3 por ciento en peso (ver composiciones de prueba 6, 3 y 5). Con un aumento adicional del contenido de agente de degradación hasta 2,7 por ciento en peso (ver composición de prueba 4), el volumen del filtrado aumentó ligeramente hasta 10,2 mL. En forma sorprendente, se observó efecto sinérgico cuando se utilizó una mezcla como el aditivo (ver composición de prueba 7). El lodo conteniendo este aditivo dio lugar a

una pérdida de fluido de solamente 6,5 mL, más bajo que el logrado con cualesquiera otras composiciones de prueba no mezcladas y muy por debajo del valor aceptable de 10 mL. Sin desear ceñirse a ninguna teoría, se cree que la sinergia se debió probablemente a la distribución física de las partículas de microgel polimérico contenidas en el lodo, las cuales mejoraron la reducción de pérdida de fluido.

Por lo tanto, la presente invención está bien adaptada para lograr los objetivos y ventajas mencionados, así como también aquellos que son inherentes a ella. Las formas particulares descritas anteriormente son ilustrativas solamente ya que la presente invención puede ser modificada y llevada a la práctica en formas diferentes pero equivalentes evidentes para los expertos en el arte que tienen el beneficio de los principios descritos aquí. Además, no se pretende establecer limitaciones con respecto a los detalles de construcción o diseño ilustrado aquí, diferentes a los descritos en las reivindicaciones finales. Por lo tanto, es evidente que las formas ilustrativas particulares descritas anteriormente pueden ser alteradas o modificadas y todas esas variaciones se consideran dentro de los alcances y espíritu de la presente invención. Todos los números y escalas descritos anteriormente pueden variar en cierta magnitud. Siempre que se describe una escala numérica

con un límite inferior y un límite superior, cualquier número y cualquier escala incluida caen dentro de la escala específicamente descrita. En particular, cada escala de valores (o sea, "desde más o menos a hasta más o menos b" o, de manera equivalente, "desde aproximadamente a hasta b" o, de manera equivalente, "desde aproximadamente a-b") descrita aquí se entenderá como indicando cada número y escala abarcados dentro de la escala más amplia de valores. Además, los artículos indefinidos "un" o "una", tal como se emplean en las reivindicaciones, están definidos aquí para significar uno o más de uno de los elementos que presenta. Del mismo modo, los términos en las reivindicaciones tienen su significado simple y corriente, a menos que el propietario de la patente lo define explícita y claramente de otro modo.

REIVINDICACIONES

1. Un lodo de perforación que contiene un fluido base acuoso y un aditivo para el control de fluido que comprende por lo menos un microgel polimérico.
2. El lodo de perforación, de acuerdo con la reivindicación 1, en el cual el aditivo para el control de pérdida de fluido está contenido en el lodo de perforación en una cantidad hasta de más o menos 5% en peso del componente activo del microgel polimérico.
3. El lodo de perforación, de acuerdo con la reivindicación 1, en el cual el microgel polimérico tiene un diámetro en la escala de más o menos 10 mm a más o menos 1 mm.
4. El lodo de perforación, de acuerdo con la reivindicación 1, en el cual el microgel polimérico comprende un producto formado mediante una reacción de polimerización de un polímero o copolímero y un agente de degradación.
5. El lodo de perforación, de acuerdo con la reivindicación 4, en el cual el polímero o copolímero comprende por lo menos una unidad basa en por lo menos un compuesto seleccionado a partir del grupo que consiste de acrilamidas, alcoholes vinílicos, vinil pirrolidonas, vinilpiridinas, acrilatos, poliacrilamidas, alcoholes polivinílicos, polivinil pirrolidonas, polivinil

piridinas, poliacrilatos, polibutileno succinato, polibutileno succinato-coadipato, polihidroxi butirato-valerato, polihidroxi butirato-covalerato, policaprolactonas, amidas poliéster, tereftalatos de polietileno, tereftalato de polietileno sulfonatado, óxidos de polietileno, polietilenos, polipropilenos, copoliéster aromático alifático, ácidos poliacrílicos, polisacáridos (tales como dextran o celulosa), quitinas, quitosanos, proteínas, poliésteres alifáticos, ácidos polilácticos, poli(glicolidos), poli(ϵ -caprolactonas), éteres poli(hidroxi éster), poli(hidroxi butiratos), poli(anhídridos), policarbonatos, poli(orto ésteres), ácidos (poliamino), óxidos poli(etileno), óxidos (polipropileno), poli(fosfazenos), amidas poliéster, poliamidas, poli-estirenos, un copolímero de los mismos, un homopolímero de los mismos, un tetrapolímero de los mismos y cualquier derivado de ellos.

6. El lodo de perforación, de acuerdo con la reivindicación 4, en el cual el agente de degradación comprende por lo menos un agente seleccionado a partir del grupo que consiste de: dietileno glicol dimetacrilato, azobisisobutironitrilo y 2,2'-azobis[2-(2-imidazolin-2-il)propano]dihidrocloruro.

7. El lodo de perforación, de acuerdo con la reivindicación 1, el cual contiene además un agente de obturación.

8. Un método que comprende:

proporcionar un fluido de tratamiento con base acuosa que contiene un aditivo para el control de pérdida de fluido que lleva por lo menos un microgel polimérico;

colocar al fluido de tratamiento con base acuosa en una formación subterránea a través de un orificio que penetra a la misma;

permitir que el aditivo para el control de pérdida de fluido quede incorporado en una torta de filtro situada sobre una superficie dentro de la formación subterránea;

permitir que la torta de filtro sea degradada; y

producir hidrocarburos a partir de la formación.

9. El método, de acuerdo con la reivindicación 8, en el cual el aditivo para el control de pérdida de fluido está incorporado en el fluido de tratamiento con base acuosa en una cantidad hasta de 5% en peso del componente activo del microgel polimérico.

10. El método, de acuerdo con la reivindicación 8, en el cual el microgel polimérico tiene un diámetro en la escala de más o menos 10 mm a más o menos 1 mm.

11. El método, de acuerdo con la reivindicación 8, en el cual el microgel polimérico contiene un producto de reacción formado mediante una reacción de polimerización de un polímero o copolímero y un agente de degradación.

12. El método, de acuerdo con la reivindicación 11, en el cual el polímero o copolímero comprende por lo menos una unidad basa en por lo menos un compuesto seleccionado a partir del grupo que consiste de acrilamidas, alcoholes vinílicos, vinil pirrolidonas, vinilpiridinas, acrilatos, poliacrilamidas, alcoholes polivinílicos, polivinil pirrolidonas, polivinil piridinas, poliacrilatos, polibutileno succinato, polibutileno succinato-coadipato, polihidroxi butirato-valerato, polihidroxi butirato-covalerato, policapro lactonas, amidas poliéster, tereftalatos de polietileno, tereftalato de polietileno sulfonatado, óxidos de polietileno, polietilenos, polipropilenos, copoliéster aromático alifático, ácidos poliacrílicos, polisacáridos (tales como dextran o celulosa), quitinas, quitosanos, proteínas, poliésteres alifáticos, ácidos polilácticos, poli(glicolidos), poli(ϵ -caprolactonas), éteres poli(hidroxi éster), poli(hidroxi butiratos), poli(anhídridos), policarbonatos, poli(orto ésteres), ácidos (poliamino), óxidos poli(etileno), óxidos (polipropileno), poli(fosfazenos), amidas poliéster, poliamidas, poli-estirenos, un copolímero de los mismos, un homopolímero de los mismos, un tetrapolímero de los mismos, derivados y cualquier combinación de ellos.

13. El método, de acuerdo con la reivindicación 11, en el cual el agente de degradación comprende por lo menos

un agente seleccionado a partir del grupo que consiste de: 2,2'-azobis-(2-metil butironitrilo), 2,2'-azobis (isobutiramida hidrocioruro), 2,2'-azobis[2-imidazolin-2-il)propano]dihidrocioruro, 1,1'-azobis(ciclohexanol carbonitrilo), 2,2'-azobis(2-metil propionamidina dihidrocioruro, ácido 4,4'-azobis(4-cianovalérico), persulfato de amonio, dihidrato de la sal monosódica de ácido hidroxí metanosulfínico, persulfato de potasio, persulfato de sodio, peróxido de benzoilo, 1,1-bis(amil peroxi terciario)ciclohexano, 1,1-bis(butilperoxi terciario) ciclohexano, 2,2-bis(butilperoxi terciario) ciclo hexano, 2,2-bis(butilperoxi terciario)butano, peróxido de 2,4-pentanodiona, 2,5-bis(butilperoxi terciario)-2,5-dimetil hexano, 2,5-di(butil peroxi terciario)-2,5-dimetil-3-hexino; peróxido de 2-butanona, hidroperóxido de cumeno, di-terciario-amil peróxido, dicumil peróxido, lauroil peróxido, hidroperóxido butílico terciario, peracetato de butilo terciario, peróxido de butilo terciario, peroxibenzoato de butilo terciario, butilperoxi terciario-2-etilhexil carbonato, dietileno glicol dimetacrilato, azobis-isobutironitrilo, derivados y sus combinaciones.

14. El método, de acuerdo con la reivindicación 8, en el cual el fluido de tratamiento con base acuosa comprende además un agente de obturación.

15. Un método que comprende:

proporcionar un lodo de perforación que contiene un fluido base acuoso y un aditivo para el control de la pérdida de fluido que contiene por lo menos un microgel polimérico;

hacer circular al lodo de perforación en una formación subterránea;

permitir que el aditivo para controlar la pérdida de fluido quede incorporado en una torta de filtro situada sobre una superficie dentro de la formación subterránea;

permitir que la torta de filtro sea degradada; y

producir hidrocarburos desde la formación.

16. El método, de acuerdo con la reivindicación 15, en el cual el aditivo para controlar la pérdida de fluido está incorporado en el lodo de perforación en una cantidad de hasta más o menos 5% en peso del componente activo del microgel polimérico.

17. El método, de acuerdo con la reivindicación 15, en el cual el microgel polimérico tiene un diámetro en la escala de más o menos 10 mm a más o menos 1 mm.

18. El método, de acuerdo con la reivindicación 15, en el cual el microgel polimérico comprende un producto de reacción formado mediante una reacción de polimerización de un polímero o copolímero y un agente de degradación.

19. El método, de acuerdo con la reivindicación 18, en el cual el polímero o copolímero comprende por lo menos una unidad basada en por lo menos un compuesto

seleccionado a partir del grupo que consiste de acrilamidas, alcoholes vinílicos, vinyl pirrolidonas, vinilpiridinas, acrilatos, poliacrilamidas, alcoholes polivinílicos, polivinil pirrolidonas, polivinil piridinas, poliacrilatos, polibutileno succinato, polibutileno succinato-coadipato, polihidroxi butirato-valerato, polihidroxi butirato-covalerato, policapro lactonas, amidas poliéster, tereftalatos de polietileno, tereftalato de polietileno sulfonatado, óxidos de polietileno, polietilenos, polipropilenos, copoliéster aromático alifático, ácidos poliacrílicos, polisacáridos (tales como dextran o celulosa), quitinas, quitosanos, proteínas, poliésteres alifáticos, ácidos polilácticos, poli(glicolidos), poli(ϵ -caprolactonas), éteres poli(hidroxi éster), poli(hidroxi butiratos), poli(anhídridos), policarbonatos, poli(orto ésteres), ácidos (poliamino), óxidos poli(etileno), óxidos (polipropileno), poli(fosfazenos), amidas poliéster, poliamidas, poli-estirenos, un copolímero de los mismos, un homopolímero de los mismos, un tetrapolímero de los mismos, derivados y cualquier combinación de ellos.

20. El método, de acuerdo con la reivindicación 18, en el cual el agente de degradación comprende por lo menos un agente seleccionado a partir del grupo que consiste de: 2,2'-azobis-(2-metil butironitrilo), 2,2'-azobis(isobutiramida hidrocioruro), 2,2'-azobis[2-imidazolin-2-

il)propano]dihidrocloruro, 1,1'-azobis(ciclohexanol carbonitrilo), 2,2'-azobis(2-metil propionamidina dihidrocloruro, ácido 4,4'-azobis(4-cianovalérico), persulfato de amonio, dihidrato de la sal monosódica de ácido hidroximetanosulfinico, persulfato de potasio, persulfato de sodio, peróxido de benzoilo, 1,1-bis(amilperoxi terciario)ciclohexano, 1,1-bis(butilperoxi terciario)ciclohexano, 2,2-bis(butilperoxi terciario)ciclohexano, 2,2-bis(butilperoxi terciario)butano, peróxido de 2,4-pentanodiona, 2,5-bis(butilperoxi terciario)-2,5-dimetilhexano, 2,5-di(butilperoxi terciario)-2,5-dimetil-3-hexino; peróxido de 2-butanona, hidroperóxido de cumeno, di-terciario-amilperóxido, dicumilperóxido, lauroilperóxido, hidroperóxido butílico terciario, peracetato de butilo terciario, peróxido de butilo terciario, peroxibenzoato de butilo terciario, butilperoxi terciario-2-etilhexil carbonato, dietileno glicol dimetacrilato, azobis-isobutironitrilo, derivados y sus combinaciones.

21. El método, de acuerdo con la reivindicación 15, en el cual el lodo de perforación comprende además un agente de obturación.

22. Un método que comprende:

proporcionar una pastilla de tamiz interno que contiene un fluido base acuoso y un aditivo para el

control de pérdida de fluido que lleva por lo menos un microgel polimérico; y

colocar a la pastilla de tamiz interno en una formación subterránea, de modo que la misma quede colocada entre un tamiz de relleno de grava y un área de la formación subterránea.

23. El método, de acuerdo con la reivindicación 22, en el cual el aditivo para controlar la pérdida de fluido está incorporado en la pastilla de tamiz interno en una cantidad de más o menos 1% en peso a más o menos 10% en peso del componente activo del microgel polimérico.

24. El método, de acuerdo con la reivindicación 22, en el cual el microgel polimérico tiene un diámetro en la escala de más o menos 10 mm a más o menos 1 mm.

25. El método, de acuerdo con la reivindicación 22, en el cual el microgel polimérico contiene un producto de reacción formado mediante una reacción de polimerización de un polímero o un copolímero y un agente de degradación.

26. El método, de acuerdo con la reivindicación 25, en el cual el agente de degradación comprende por lo menos un agente seleccionado a partir del grupo que consiste de: 2,2'-azobis-(2-metil butironitrilo), 2,2'-azobis (isobutiramida hidrocloreuro), 2,2'-azobis[2-imidazolin-2-il)propano]dihidrocloreuro, 1,1'-azobis(ciclohexanol carbonitrilo), 2,2'-azobis(2-metil propionamidina

dihidrocloruro, ácido 4,4'-azobis(4-cianovalérico), persulfato de amonio, dihidrato de la sal monosódica de ácido hidroximetanosulfínico, persulfato de potasio, persulfato de sodio, peróxido de benzoilo, 1,1-bis(amilperoxi terciario)ciclohexano, 1,1-bis(butilperoxi terciario)ciclohexano, 2,2-bis(butilperoxi terciario)ciclohexano, 2,2-bis(butilperoxi terciario)butano, peróxido de 2,4-pentanodiona, 2,5-bis(butilperoxi terciario)-2,5-dimetilhexano, 2,5-di(butilperoxi terciario)-2,5-dimetil-3-hexino; peróxido de 2-butanona, hidroperóxido de cumeno, di-terciario-amilperóxido, dicumilperóxido, lauroilperóxido, hidroperóxido butílico terciario, peracetato de butilo terciario, peróxido de butilo terciario, peroxibenzoato de butilo terciario, butilperoxi terciario-2-etilhexil carbonato, dietileno glicol dimetacrilato, azobis-isobutironitrilo, derivados y sus combinaciones.

27. El método, de acuerdo con la reivindicación 25, en el cual el polímero o copolímero comprende por lo menos una unidad basada en por lo menos un compuesto seleccionado a partir del grupo que consiste de: acrilamidas, alcoholes vinílicos, vinilpirrolidonas, vinilpiridinas, acrilatos, poliacrilamidas, alcoholes polivinílicos, polivinilpirrolidonas, polivinilpiridinas, poliacrilatos, polibutileno succinato, polibutileno succinato-coadipato, polihidroxi butirato-

valerato, polihidroxi butirato-covalerato, policaprolactonas, amidas poliéster, tereftalatos de polietileno, tereftalato de polietileno sulfonatado, óxidos de polietileno, polietilenos, polipropilenos, copoliéster aromático alifático, ácidos poliacrílicos, polisacáridos (tales como dextran o celulosa), quitinas, quitosanos, proteínas, poliésteres alifáticos, ácidos polilácticos, poli(glicolidos), poli(ϵ -caprolactonas), éteres poli(hidroxi éster), poli(hidroxi butiratos), poli(anhídridos), policarbonatos, poli(orto ésteres), ácidos (poliamino), óxidos poli(etileno), óxidos (polipropileno), poli(fosfazenos), amidas poliéster, poliamidas, poli-estirenos, un copolímero de los mismos, un homopolímero de los mismos, un tetrapolímero de los mismos, derivados y cualquier combinación de ellos.

**MÉTODOS PARA EL USO DE ADITIVOS DE PÉRDIDA DE FLUIDO QUE
CONTIENEN MICROGELES**

Resumen

Se proporciona métodos y fluidos que incluyen pero no están limitados a un lodo de perforación que contiene un fluido base acuoso y un aditivo para controlar la pérdida de fluido que contiene por lo menos un microgel polimérico; y un método que comprende: proporcionar un fluido de tratamiento con base acuosa que contiene un aditivo para controlar la pérdida de fluido que comprende por lo menos un microgel polimérico; colocar al fluido de tratamiento con base acuosa en una formación subterránea a través de un orificio de pozo que penetra a esta última; permitir que el aditivo para el control de pérdida de fluido quede incorporado en una torta de filtro situada sobre una superficie dentro de la formación subterránea; permitir que la torta de filtro sea degradada y producir hidrocarburos desde la formación. Se proporciona también métodos adicionales.

La figura 1 es una fotografía de una solución transparente de una dispersión de microgel después de disolución en agua

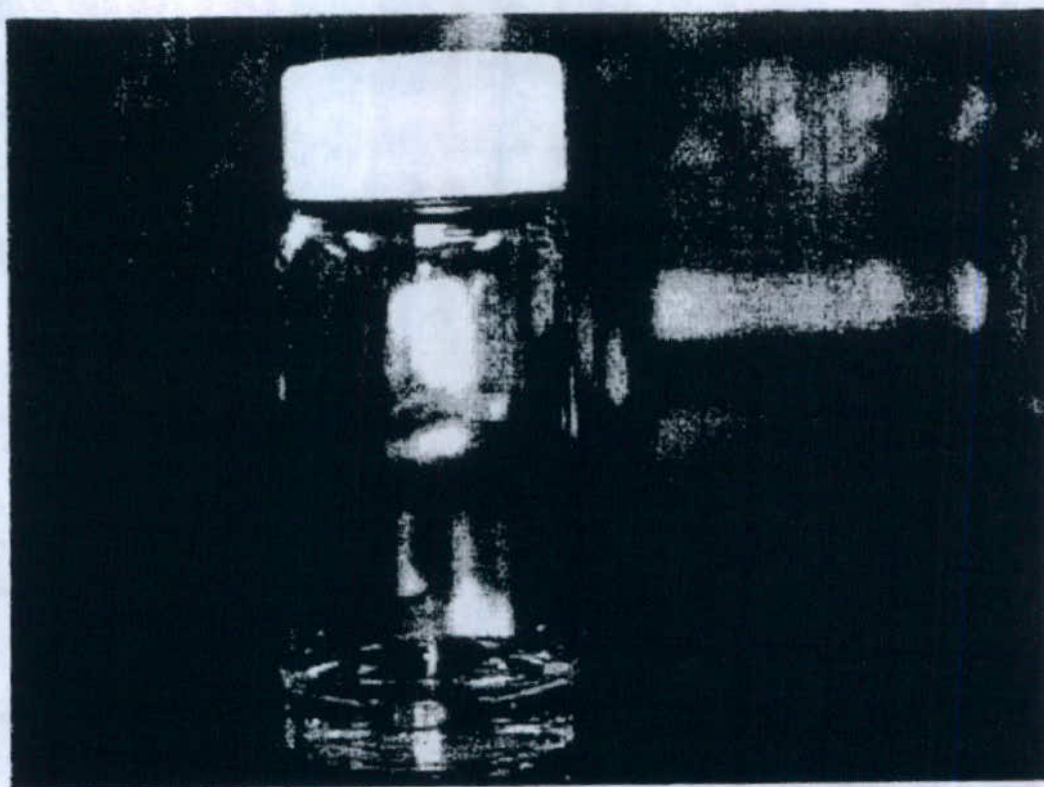


Figure 1

La figura 2 es una micrografía de la dispersión de
microgel de la figura 1

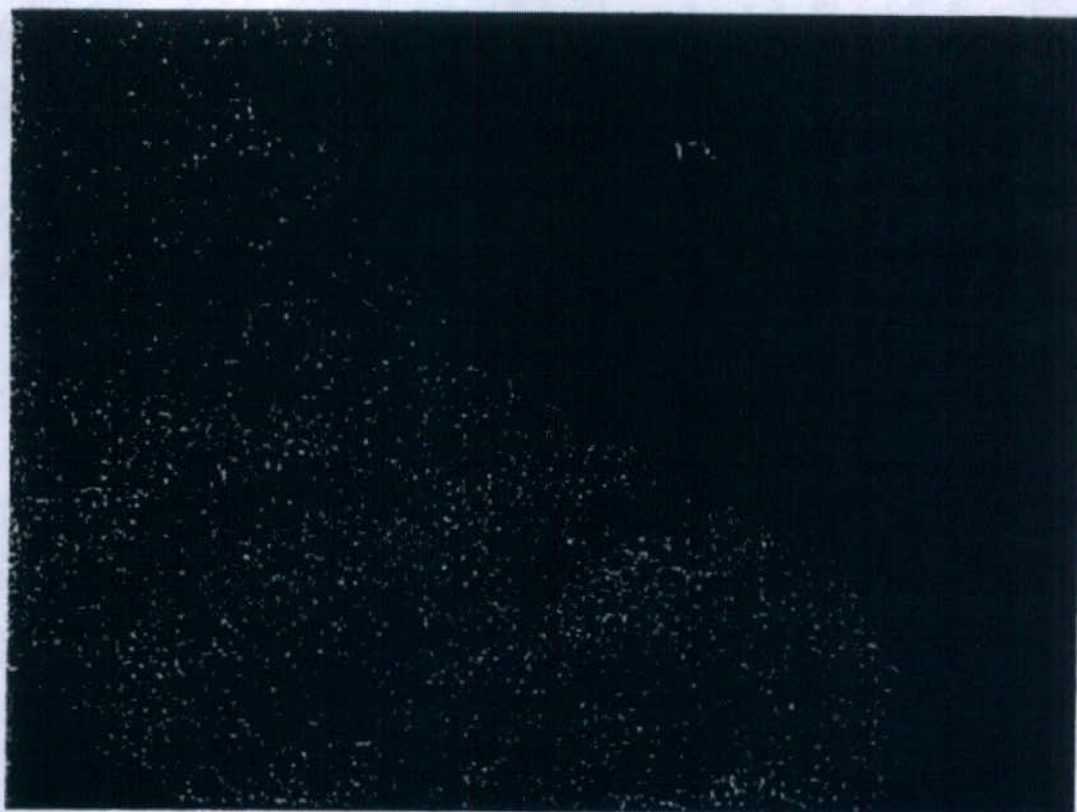


Figure 2

METHODS OF USING FLUID LOSS ADDITIVES COMPRISING MICRO GELS

BACKGROUND

[0001] The present invention relates to methods of using fluid loss control additives. More specifically, at least in some embodiments, the present invention relates to the use of fluid loss control additives in drilling and servicing fluids that comprise polymeric micro gels in subterranean operations.

[0002] When well bores are drilled into producing formations, drilling fluids are utilized which will minimize damage to the permeability of the formations and their ability to produce hydrocarbons. Servicing fluids are utilized when completion operations are conducted in producing formations and when conducting work-over operations in the formations. The drilling and servicing fluids deposit a layer of particles known as "filter cake" on the walls of the well bores within the producing formations. The filter cake is believed to help prevent the drilling and servicing fluids from being lost into the formation and prevents solids from entering the porosities of the rock. Following completion and prior to initiating production, the filter cake is usually degraded or allowed to degrade to allow product to flow into the well bore for production. Degrading the filter cake is important to retain well bore connectivity and the natural permeability of the reservoir rock. If not degraded or allowed to degrade, the filter cake could present an impediment to production, *inter alia*, by altering the permeability of the reservoir. Once the permeability of the reservoir has been diminished, it is seldom able to restore it to its original condition. These should be distinguished from the function of additives (sometimes termed in the art "relative permeability modifiers" or "RPMs") that are often used in conformance or fracturing fluids to permanently seal water influxes to hydrocarbon reservoir areas surrounding a well bore.

[0003] Drilling and servicing fluids (such as drill-in fluids) may comprise fluid loss control additives to further assist in preventing the drilling and servicing fluids from being lost into the formations. Drilling fluids are any of a number of fluids and mixtures of fluids and solids (as solid suspensions, mixtures and emulsions of liquids, gases and solids) used in operations to drill boreholes into the earth. Classifications of drilling fluids has been attempted in many ways, often producing more confusion than insight. One classification scheme, given here, is based only on the mud composition by singling out the component that clearly defines the function and performance of the fluid: (1) aqueous-based, (2) oil-based and (3) gaseous

(pneumatic). Each category has a variety of subcategories that overlap each other considerably. Ideally, a drilling fluid is non-damaging to the formation, meaning that the fluid does not leave behind particulates, fines, etc. that negatively impact the permeability of the formation.

[0004] A drill-in fluid is a fluid designed for drilling through the reservoir section of a well bore in a subterranean formation. The reasons for using a specially designed fluid include, but are not necessarily limited to: (1) to drill the reservoir zone successfully, often a long, horizontal drain hole; (2) to minimize damage to and maximize production of exposed zones; and (3) to facilitate the well completion needed, which may include complicated procedures. A drill-in fluid often resembles a completion fluid in that it may comprise a brine, possibly bridging agents, and/or polymers.

[0005] The term "drilling fluid" as used herein refers generically to both drilling fluids and drill-in fluids unless otherwise specified.

[0006] Other types of treatment fluids that can utilize fluid loss control materials include, but are not limited to, pills (such as inside screen pills), which are fluids with a relatively small quantity (*e.g.*, less than 200 bbl) of a special blend of drilling fluid to accomplish a specific task that the regular drilling fluid cannot perform. Examples include high-viscosity pills to help lift cuttings out of a vertical well bore, freshwater pills to dissolve encroaching salt formations, pipe-freeing pills to destroy filter cake and relieve differential sticking forces, and lost circulation material pills to plug a thief zone. Another example is a screen pill that may be useful in conjunction with a gravel pack operation.

[0007] Examples of conventional fluid loss control additives for water-based treatment fluids include nonionic water soluble polymers, such as starches, derivatized starches, gums, derivatized gums, and cellulose. Fluid loss additives that include starches often vary in the ratio of amylose to amylopectin content, and may or may not be modified with a crosslinking agent such as epichlorohydrin. Also, natural starches may not be uniform in terms of quality and effectiveness. These cross-linked starches often do not have thermal stability at temperatures up to about 250 °F, and at temperatures above 250 °F, they can only effectively be used by increased loading the treatment fluid with the cross-linked starch, constantly replenishing the treatment fluid with the cross-linked starch, or using an oxygen scavenger in conjunction with the cross-linked starch. At temperatures above 300 °F, even by the use of the above mentioned measures, cross-linked starches may not be effective fluid loss control additives.

[0008] Conventional linear synthetic polymers are also utilized, but oftentimes, they require another additive, such as a clay, to be able to effectively function as fluid loss control additives. However, the use of clay can be problematic in drill-in fluids, as removing the clay from the subterranean formation can be difficult because it infiltrates into pores of the subterranean formation. Furthermore, the addition of clay to a treatment fluid dramatically increases the viscosity of the fluid, which can cause drilling to be completed at a reduced rate. Another issue is that some synthetic polymers cannot be used successfully in conjunction with some brines, for instance, divalent brines. This brine incompatibility is thought to prevent the synthetic polymer from associating with the clay to forming bridging colloids, which are desirable because they provide a degree of fluid loss, *e.g.*, in a drill-in fluid.

SUMMARY

[0009] The present invention relates to methods of using fluid loss control additives. More specifically, at least in some embodiments, the present invention relates to the use of fluid loss control additives in drilling and servicing fluids that comprise polymeric micro gels in subterranean operations.

[0010] In one embodiment, the present invention provides a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel.

[0011] In another embodiment, the present invention provides a method comprising: providing an aqueous based treatment fluid comprising a fluid loss control additive that comprises at least one polymeric micro gel; placing the aqueous based treatment fluid in a subterranean formation via a well bore penetrating the subterranean formation; allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation; allowing the filter cake to be degraded; and producing hydrocarbons from the formation.

[0012] In another embodiment, the present invention provides a method comprising: providing a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel; circulating the drilling fluid in a subterranean formation; allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation; allowing the filter cake to be degraded; and producing hydrocarbons from the formation.

[0013] In another embodiment, the present invention provides a method comprising: providing an inside screen pill comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel, and placing the inside screen pill in a subterranean formation so that the inside screen pill is located between a gravel pack screen and an area of the subterranean formation.

[0014] The features and advantages of the present invention will be readily apparent to those skilled in the art. While numerous changes may be made by those skilled in the art, such changes are within the spirit of the invention.

BRIEF DESCRIPTION OF THE DRAWINGS

[0015] These drawings illustrate certain aspects of some of the embodiments of the present invention, and should not be used to limit or define the invention.

[0016] Figure 1 is a photograph of a transparent solution of a micro gel dispersion after dissolving in water.

[0017] Figure 2 is a micrograph of the micro gel dispersion of Figure 1.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0018] The present invention relates to methods of using fluid loss control additives. More specifically, at least in some embodiments, the present invention relates to the use of fluid loss control additives in drilling and servicing fluids that comprise polymeric micro gels in subterranean operations.

[0019] There may be several potential advantages to the methods and compositions of the present invention, only some of which are alluded to herein. The present application provides drilling fluids, preferably for use as a drilling, drill-in, completion fluid or pills, which quickly forms a thin, degradable filter cake. Again, for the sake of clarity, the term "drilling fluid" as used herein refers generically to both drilling fluids and drill-in fluids unless otherwise specified. The drilling fluids of the present invention are less invasive to the formation yet provide lubricity, effective fluid loss control, and good filter cake sealing and plastering characteristics. The resulting filter cake can be easily lifted off and readily removed or dissolved to help ensure low skin and residual damage. Moreover, the fluids are stable for prolonged periods of time at high temperatures. In particular, the drilling fluids of the present invention are believed to have enhanced performance over conventional fluids that comprise starch derivatives, for instance, because they can be used at effective static bottom hole temperatures up to at least about 400 °F or more while maintaining effective fluid loss control, thus minimizing formation damage. In doing so, a vast range of applications is open for technological advancement and new product revenue. It is believed that in equal concentrations, the drilling fluids of the present invention that comprise fluid loss additives comprising polymeric micro gels described herein give an increased performance (*e.g.*, an order of magnitude increase) in the amount of fluid loss over starch-based fluid loss materials.

[0020] Another potential advantage may be that the drilling fluids of the present invention may be used without the addition of clay, which allows for easier clean up, minimal formation damage, faster drilling times, and the avoidance of problems associated with the increased viscosity of fluids when clay is added.

[0021] In some embodiments, the present invention provides an aqueous-based drilling fluid that comprises a fluid loss control additive of the present invention that comprises at least one polymeric micro gel. In other embodiments, the present invention provides drilling fluids that comprise an aqueous base fluid (*e.g.*, a brine or fresh water), and one or more fluid

loss control additives that comprise at least one polymeric micro gel. In practical usage, with respect to building a filter cake that will easily degrade, it may be beneficial to include bridging agents in the drilling fluid that will become incorporated into the filter cake. Without the bridging agents, the polymeric micro gels may form an effective filter cake, but that filter cake may be more difficult to remove. Other components of the drilling fluid may comprise weighting agents, clays, polymers, as well as other additives common to aqueous-based drilling fluids including, but not necessarily limited to, lubricants, corrosion inhibitors, other inhibitors, and oxygen scavengers.

[0022] In some embodiments, the fluid loss control additive may be included in the drilling fluid in an amount of about 5% or below by weight of active component of the polymeric micro gel.

[0023] The base fluid of the drilling fluids of the present invention may be any suitable aqueous-based fluid including, but not necessarily limited to, brines (*i.e.*, water comprising a salt). In some instances, brines having a density of at least about 9 lb/gal, referred to as "high density brines," may be suitable. The brines may contain substantially any suitable salts, including, but not necessarily limited to, salts based on metals, such as calcium, magnesium, sodium, potassium, cesium, zinc, aluminum, and lithium. Salts of calcium and zinc are preferred. The salts may contain substantially any anions, with preferred anions being less expensive anions including, but not necessarily limited to chlorides, bromides, formates, acetates, and nitrates. Generally, the base fluid is present in a drilling fluid of the present invention in an amount in the range of from about 35% to about 97% by weight thereof, more preferably from about 70% to about 95% or more.

[0024] Preferably, when used, the bridging agents are either self-degrading or degradable in a suitable clean-up solution (*e.g.*, a mutual solvent, water, an acid solution, etc.). Examples of bridging agents suitable for use in the methods of the current invention include, but are not necessarily limited to, magnesium citrate, calcium citrate, calcium succinate, calcium maleate, calcium tartrate, magnesium tartrate, bismuth citrate, calcium carbonate, sodium chloride and other salts, and the hydrates thereof. Examples of degradable bridging agents may include, but are not necessarily limited to, bridging agents comprising degradable materials such as degradable polymers. A polymer is considered to be "degradable" herein if the degradation is due to, *inter alia*, chemical and/or radical process such as hydrolysis, oxidation, enzymatic

degradation, or UV radiation. Suitable examples of degradable polymers that may be used in accordance with the present invention include, but are not necessarily limited to, those described in the publication of *Advances in Polymer Science*, Vol. 157 entitled "Degradable Aliphatic Polyesters" edited by A. C. Albertsson, the disclosure of which is hereby incorporated by reference. Specific examples of suitable polymers include, but are not necessarily limited to, polysaccharides such as dextrans or celluloses; chitins; chitosans; proteins; orthoesters; aliphatic polyesters; poly(lactides); poly(glycolides); poly(ϵ -caprolactones); poly(hydroxybutyrates); poly(anhydrides); aliphatic polycarbonates; poly(orthoesters); poly(amino acids); poly(ethylene oxides); and polyphosphazenes. Combinations and derivatives of these are suitable as well.

[0025] When choosing a particular bridging agent to use, one should be aware of the performance of that bridging agent at the temperature range of the application. The bridging agents utilized may be generally present in the drilling fluid compositions in an amount in the range of from about 1% to about 40% by weight thereof, more preferably from about 5% to about 25%. Generally, the bridging agents may have a particle size in the range of from about 1 micron to about 600 microns. Preferably, the bridging particle size is in the range of from about 1 to about 200 microns but may vary from formation to formation. The particle size used is determined by the pore throat size of the formation.

[0026] The term "polymeric micro gel" as used herein refers to a gelled particle comprising a cross-linked polymer (*e.g.*, water-soluble and water-swellaable) network. In some embodiments, the fluid loss control additive may be delivered in a continuous medium, *e.g.*, in a dispersion form. In other embodiments, the polymeric micro gels may be used without any continuous medium, *e.g.*, in dry form. One of the many useful features of certain embodiments of the polymeric micro gels of the present invention is that they are believed to swell or shrink in response to an external stimuli, *e.g.*, pH and temperature. In some embodiments, some of the polymeric micro gels may be considered to be super absorbent in that they can contain over 99% water. This may be advantageous in some applications wherein water absorbance is important.

[0027] When comprising a substantial portion of the fluid loss control additive in a drilling fluid of the present invention, the polymeric micro gels optionally may be dispersed in a continuous medium before addition to the fluid. In some embodiments, the fluid loss control additive may comprise a continuous medium. Examples of suitable continuous mediums may include, but are not necessarily limited to, aqueous-based fluids, alcohols, glycerin, glycols,

polyglycol amines, polyols, and any derivative thereof. Additionally, in some embodiments, the continuous medium may comprise a fluid selected from the group consisting of methanol, ethanol, *n*-propanol, isopropanol, *n*-butanol, *sec*-butanol, isobutanol, *t*-butanol, a mixture of methanol, ethanol, *n*-propanol, isopropanol, *n*-butanol, *sec*-butanol, isobutanol, or *t*-butanol and water, a mixture of ammonium sulfate, sodium sulfate, or potassium sulfate and water, a mixture of sodium chloride, potassium chloride, or calcium chloride and water, and combinations thereof. Optionally, the continuous medium comprises a fluid selected from the group consisting of ethanol, a mixture of *t*-butanol and water, and a mixture of ammonium sulfate and water. Mixtures of these may be suitable as well. Examples of suitable aqueous-based fluids may include, but are not necessarily limited to, fresh water, sea water, salt water, and brines (*e.g.*, saturated salt waters). Examples of suitable brines may include, but are not necessarily limited to, heavy brines, monovalent brines, divalent brines, and trivalent brines that comprise a soluble salts like sodium chloride, calcium chloride, calcium bromide, zinc bromide, potassium carbonate, sodium formate, potassium formate, cesium formate, sodium acetate, potassium acetate, calcium acetate, ammonium acetate, ammonium chloride, ammonium bromide, sodium nitrate, potassium nitrate, ammonium nitrate, ammonium sulfate, calcium nitrate, sodium carbonate, potassium carbonate, any combination thereof, and any derivative thereof. Examples of suitable alcohols may include, but are not necessarily limited to, methanol, ethanol, propanol, iso-propanol, butanol, tert-butanol, and the like. Examples of suitable glycols may include, but are not necessarily limited to, polyglycols, propylene glycol, ethylene glycol, and the like. In some embodiments, the continuous medium may be present in any suitable range including ratios of continuous medium to polymeric micro gel of about 30:70, about 20:80, about 10:90, about 5:95, and about 0:100 by weight.

[0028] In some embodiments, the polymeric micro gels may comprise a reaction product formed by a suitable polymerization reaction of a polymer (or monomer) and a crosslinking agent. In one embodiment of a method of forming the micro gels of the present invention, the polymerization is a dispersion polymerization in a continuous medium that is substantially inert toward chain transfer reactions. Crosslinked polymer micro gel particles are formed, which are believed to be insoluble or at most swellable in the continuous medium. While not wishing to be limited to any particular theory, it is believed that in some embodiments, the crosslinking agent may act as an initiator forming chain branches of the polymer or

monomer, which in turn may react with one another to form a polymeric micro gel. Other suitable initiators may include those known to those skilled in the art such as photoinitiators, thermal initiators, and combinations thereof.

[0029] In some embodiments, the polymer (or monomer) may comprise any suitable polymer or monomer that can form a polymeric micro gel when crosslinked. Suitable polymers and monomers may include, but are not necessarily limited to, those that comprise units based on acrylamides, vinyl alcohols, vinylpyrrolidones, vinylpyridines, acrylates, polyacrylamides, polyvinyl alcohols, polyvinylpyrrolidones, polyvinylpyridines, polyacrylates, polybutylene succinate, polybutylene succinate-co-adipate, polyhydroxybutyrate-valerate, polyhydroxybutyrate-covalerate, polycaprolactones, polyester amides, polyethylene terephthalates, sulfonated polyethylene terephthalate, polyethylene oxides, polyethylenes, polypropylenes, aliphatic aromatic copolyester, polyacrylic acids, polysaccharides (such as dextran or cellulose), chitins, chitosans, proteins, aliphatic polyesters, polylactic acids, poly(glycolides), poly(ϵ -caprolactones), poly(hydroxy ester ethers), poly(hydroxybutyrates), poly(anhydrides), polycarbonates, poly(orthoesters), poly(amino acids), poly(ethylene oxides), poly(propylene oxides), poly(phosphazenes), polyester amides, polyamides, polystyrenes, any derivative thereof, any copolymer, homopolymer, or terpolymer, or any blend thereof. In some embodiments, the monomer may include an unsaturated group, such as a monomer including a vinyl group. Exemplary vinyl-containing monomers may be described by the formula $C(R_1)(R_2)=C(R_3)(R_4)$, wherein R_1 , R_2 , R_3 and R_4 are segments rendering the solubility or swellability of this monomer in the common solvent. Optionally, R_1 , R_2 , R_3 and R_4 can each be independently selected from, but not limited to, hydrogen, methyl, ethyl, $CONH_2$, $CONHCH_3$, $CON(CH_3)_2$, CH_2SO_3H , CH_2SO_3Na and $COONa$. In a further option, the monomer comprises a compound selected from the group consisting of hydroxyethyl acrylate, acrylamide and hydroxyethyl methacrylate. Examples of suitable copolymers that may be used with these may include inorganic and organic polymers. Some of these polymers and monomers may be more suited for less high temperature applications than others; however, crosslinking may increase their thermal stability. In some embodiments the polymer or monomer may comprise a water soluble polymer or monomer. In some embodiments the polymer or monomer may comprise a block copolymer with a portion that is water soluble and a portion that is water insoluble.

[0030] In some embodiments, the crosslinking agent may comprise any crosslinking agent that can react with a polymer or monomer to form a polymeric micro gel. In some embodiments, the crosslinking agent may comprise a covalent crosslinking agent. Examples of suitable crosslinking agents may include crosslinking agents that form a radical. Suitable crosslinking agents may include, but are not limited to, 2,2'-azobis-(2-methylbutyronitrile), 2,2'-azobis(isobutyramidine hydrochloride), 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride, 1,1'-azobis(cyclohexanecarbonitrile), 2,2'-azobis(2-methylpropionamidine) dihydrochloride, 4,4'-azobis(4-cyanovaleric acid), ammonium persulfate, hydroxymethanesulfinic acid monosodium salt dihydrate, potassium persulfate, sodium persulfate, benzoyl peroxide, 1,1-bis(*tert*-amylperoxy)cyclohexane, 1,1-bis(*tert*-butylperoxy)cyclohexane, 2,2-bis(*tert*-butylperoxy)butane, 2,4-pentanedione peroxide, 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane, 2,5-di(*tert*-butylperoxy)-2,5-dimethyl-3-hexyne; 2-butanone peroxide, cumene hydroperoxide, di-*tert*-amyl peroxide, dicumyl peroxide, lauroyl peroxide, *tert*-butyl hydroperoxide, *tert*-butyl peracetate, *tert*-butyl peroxide, *tert*-butyl peroxybenzoate, *tert*-butylperoxy-2-ethylhexyl carbonate, diethylene glycol dimethacrylate, azobisisobutyronitrile, and combinations thereof. A person of ordinary skill in the art would be able to select an appropriate crosslinking agent based upon the polymer or monomer to be crosslinked.

[0031] In some embodiments, the amount of crosslinking agent used in conjunction with the polymer or monomer may be any amount necessary to create the desired degree of crosslinking, and other characteristics. In some embodiments, the ratio of crosslinking agent to monomer or polymer used to form the polymeric micro gel may be in the range of from about 100:1 to about 5,000:1 by weight.

[0032] In one embodiment of a method of forming the micro gels of the present invention, the polymerization is a dispersion polymerization in a continuous medium such as a continuous medium comprising *t*-butyl alcohol/water and ammonium sulfate. Both the monomer (such as, but not limited to, acrylamide) and the crosslinking agent (such as, but not limited to, *N,N'*-ethylene-bis-acrylamide) are substantially soluble or at least swellable in the continuous medium. The resultant micro gels are not soluble in the continuous medium. The continuous medium is believed to be relatively inert (or at least not so reactive as to disrupt micro gels from

forming) toward chain transfer reactions by propagating radicals. By removing the continuous medium using a suitable technique, the micro gels may be dried if desired.

[0033] Optionally, a colloidal stabilizer may also be included in the continuous medium during polymerization. One example of a colloidal stabilizer is amphiphilic, such as an amphiphilic colloidal stabilizer comprising a stabilizer selected from the group consisting of poly(vinyl pyrrolidone) (PVP), polydiallyldimethylammonium chloride (poly-DADMAC), and combinations thereof.

[0034] In one embodiment of a method of forming the micro gels of the present invention, the polymerization is a dispersion polymerization of water-soluble or swellable vinyl monomer(s) (as represented by $C(R1)(R2)=C(R3)(R4)$, where R1, R2, R3 and R4 are segments rendering the solubility or swellability of this monomer to water and the polymerization medium) in the presence of radically polymerizable crosslinking agent (such as, but not limited to, $C(R5)(R6)=C(R7)-R8-C(R9)=C(R10)(R11)$, where R5 - R11 are groups rendering solubility and swellability of this crosslinking agent to water and the polymerization medium) in a medium (such as, but not limited to, t-butanol/water and ammonium sulfate solution in water), to which the chain transfer of propagating radical is avoided or suppressed. By taking advantage of the poor stability of the resultant dispersion at high polymer content (e.g., 13 wt %), in the absence of any crosslinking agent, the result may be one chunk of macro gel.

[0035] The size range for these polymeric micro gels may range from about 10 nm to about 1 mm in diameter. A suitable size range may be 10 microns to about 500 microns. In a given composition, a range of disparate sized polymeric micro gels may be used. One of ordinary skill in the art would be able to select an appropriate size of the polymeric micro gels based upon the characteristics of the subterranean formation in which the polymeric micro gels would be used, the temperature of the subterranean formation, the degree of stability required of the polymeric micro gels, and the costs. Also, the shapes of the polymeric micro gels may vary, and may depend on several factors including, but not necessarily limited to, temperature, the continuous medium used, and the degree of crosslinking. The polymeric micro gels can be any shape, for example, at least partially spherical. Depending on the method in which the polymeric micro gels are created, the shape of the polymeric micro gels can vary. One of ordinary skill in the art would be able to select an appropriate shape of the polymeric micro gels based upon the characteristics of the subterranean formation in which the polymeric micro gels would be used,

the temperature of the subterranean formation, the degree of stability required of the polymeric micro gels, and the costs. No particular shape is critical for use in the present invention.

[0036] In some embodiments, the fluid loss control additives of the present invention may not be delivered with a continuous medium. These may be referred to as the "dry" embodiments. For example, in some embodiments, after the polymeric micro gels are polymerized, the polymeric micro gels may be removed from the continuous medium and then dried using a suitable technique to form the powdered polymeric micro gels.

[0037] In some embodiments, the present invention comprises an aqueous based drill-in fluid comprising a fluid loss control additive of the present invention that comprises at least one polymeric micro gel. In some embodiments, a fluid loss control additive may be included in the drill-in fluid in an amount of about 5% or below by weight of active component of the polymeric micro gel. Other components of the drill-in fluids may comprise bridging agents and other commonly used additives.

[0038] In some embodiments, the present invention comprises an inside screen pill comprising a water-based fluid and a fluid loss control additive comprising at least one polymeric micro gel. In some embodiments, fluid loss control additive may be included in the inside screen pill in an amount of about 1 to about 10% by weight of active component of the polymeric micro gel.

[0039] In some embodiments, the present invention provides a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel.

[0040] In some embodiments, the present invention provides a method comprising: providing an aqueous based treatment fluid comprising a fluid loss control additive that comprises at least one polymeric micro gel; and placing the aqueous based treatment fluid in a subterranean formation via a well bore penetrating the subterranean formation.

[0041] In some embodiments, the present invention provides a method comprising: providing a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel; and circulating the drilling fluid in a subterranean formation.

[0042] In some embodiments, the present invention provides a method comprising: providing an inside screen pill comprising an aqueous base fluid and a fluid loss

control additive that comprises at least one polymeric micro gel, and placing the inside screen pill in a subterranean formation so that the inside screen pill is located between a gravel pack screen and an area of the subterranean formation.

[0043] To facilitate a better understanding of the present invention, the following examples of certain aspects of some embodiments are given. In no way should the following examples be read to limit, or define, the entire scope of the invention.

EXAMPLES

Example 1

[0044] Polymeric micro gel particles were prepared in accordance with the following procedure. A mixture of 95.0 g of ethanol, 20.0 g of acrylamide, 0.50 g of poly(vinyl pyrrolidone), and 0.50 g of diethylene glycol dimethacrylate was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles with sizes from 10 μm to 100 μm (in water) was obtained with solids content of approximately 17% by weight. Figure 1 is a photograph of the transparent micro gel dispersion formed in this example. Figure 2 is a micrograph of the micro gel dispersion formed in this example. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 2

[0045] Polymeric micro gel particles were prepared in accordance with the following procedure. A mixture of 95.0 g of a mixture of t-butanol and water (90% t-butanol by weight), 15.0 g of acrylamide, 0.20 g of N,N'-ethylene-bis-acrylamide, and 0.50 g of poly(vinyl pyrrolidone) was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a

dispersion of particles with sizes from 10 μm to 300 μm was obtained with a solids content of approximately 13% by weight.

Example 3

[0046] Polymeric micro gel particles were prepared in accordance with the following procedure. A mixture of 75.5 g of a 40% solution of ammonium sulfate in water, 7.5 g of acrylamide, 12.0 g of a 20% solution of polydiallyldimethylammonium chloride in water, 6.7 μL of poly(ethylene glycol) diacrylate, and 20.0 g of de-ionized water was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 35°C. A 5.0 g solution of 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride in water (0.01% 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride by weight) was then injected into the mixture. The mixture was then kept at 35°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles with a solid content of approximately 9% by weight was obtained. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 4

[0047] Polymeric micro gel particles were prepared in accordance with the following procedure. A mixture of 90.0 g of a mixture of t-butanol and water (90% t-butanol by weight), 15.0 g of acrylamide, 0.50 g of poly(vinyl pyrrolidone), was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. After approximately 20 minutes a solution of 0.10 g of N,N'-ethylene-bis-acrylamide in 5.0 g of a mixture of t-butanol and water (90% t-butanol by weight) was injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles with sizes from 10 μm to 300 μm was obtained with a solid content of approximately 13% by weight. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then

decanted and sediments were collected. The sediments were then dried under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 5

[0048] Polymeric micro gel particles were prepared in accordance with the following procedure. A mixture of 75.5 g of a 40% solution of ammonium sulfate in water, 7.5 g of acrylamide, 12.0 g of a 20% solution of polydiallyldimethylammonium chloride in water, 67 µL of poly(ethylene glycol) diacrylate, and 20.0 g of de-ionized water was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 35°C. A 5.0 g solution of 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride in water (0.01% 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride by weight) was then injected into the mixture. The mixture was then kept at 35°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles was obtained with a solid content of approximately 9% by weight. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 6

[0049] Test composition 1 was prepared in accordance with the following procedure. A mixture of 75.5 g of a 40% solution of ammonium sulfate in water, 7.5 g of acrylamide, 12.0 g of a 20% solution of polydiallyldimethylammonium chloride in water, and 20.0 g of de-ionized water was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 35°C. A 5.0 g solution of 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride in water (0.01% 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride by weight) was then injected into the mixture. The mixture was then kept at 35°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles was obtained with a solid content of approximately 9% by weight.

Example 7

[0050] Test composition 2 was prepared in accordance with the following procedure. A mixture of 75.5 g of a 40% solution of ammonium sulfate in water, 7.5 g of

acrylamide, 12.0 g of a 20% solution of polydiallyldimethylammonium chloride in water, 6.7 μ L of poly(ethylene glycol) diacrylate, and 20.0 g of de-ionized water was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 35°C. A 5.0 g solution of 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride in water (0.01% 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride by weight) was then injected into the mixture. The mixture was then kept at 35°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles with a solid content of approximately 9% by weight was obtained.

Example 8

[0051] Test composition 3 was prepared in accordance with the following procedure. A mixture of 90.0 g of a mixture of t-butanol and water (90% t-butanol by weight), 15.0 g of acrylamide, 0.50 g of poly(vinyl pyrrolidone), was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. After approximately 20 minutes a solution of 0.10 g of N,N'-ethylene-bis-acrylamide in 5.0 g of a mixture of t-butanol and water (90% t-butanol by weight) was injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles with sizes from 10 μ m to 300 μ m was obtained with a solid content of approximately 13% by weight. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 9

[0052] Test composition 4 was prepared in accordance with the following procedure. A mixture of 90.0 g of a mixture of t-butanol and water (90% t-butanol by weight), 15.0 g of acrylamide, 0.50 g of poly(vinyl pyrrolidone), was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. After approximately

20 minutes a solution of 0.40 g of N,N'-ethylene-bis-acrylamide in 5.0 g of a mixture of t-butanol and water (90% t-butanol by weight) was injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles was obtained with a solid content of approximately 13% by weight. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 10

[0053] Test composition 5 was prepared in accordance with the following procedure. A mixture of 90.0 g of a mixture of t-butanol and water (90% t-butanol by weight), 15.0 g of acrylamide, 0.50 g of poly(vinyl pyrrolidone), was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. After approximately 20 minutes a solution of 0.20 g of N,N'-ethylene-bis-acrylamide in 5.0 g of a mixture of t-butanol and water (90% t-butanol by weight) was injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles was obtained with a solid content of approximately 13% by weight. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 11

[0054] Test composition 6 was prepared in accordance with the following procedure. A mixture of 90.0 g of a mixture of t-butanol and water (90% t-butanol by weight), 15.0 g of acrylamide, 0.50 g of poly(vinyl pyrrolidone), was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. After approximately 20 minutes a solution of 0.050 g of N,N'-ethylene-bis-acrylamide in 5.0 g of a mixture of t-

butanol and water (90% t-butanol by weight) was injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles was obtained with a solid content of approximately 13% by weight. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 12

[0055] Test composition 7 was prepared in accordance with the following procedure. Equal amounts of the powders from Examples 8, 9, 10, and 11 were mixed to generate a powdered fluid loss control additive.

Example 13

[0056] Fluid loss tests were performed on test compositions 1-7 in accordance with the following procedure. Varying dosages of each of the test compositions as well as conventional fluid loss control additive N-Drill HT Plus® (commercially available from Halliburton Energy Services, Inc., Houston, Texas) were added to separate drilling mud compositions comprising 0.09 g of viscosifier N-VIS® (commercially available from Halliburton Energy Services, Inc., Houston, Texas), 14.0 g of bridging agent BARACARB® 5, (commercially available from Halliburton Energy Services, Inc., Houston, Texas) 3.5 g of bridging agent CARACARB® 5 (commercially available from Halliburton Energy Services, Inc., Houston, Texas), and 137 g of a 26% by weight solution of NaCl in water. The amount of NaCl in the drilling fluids that the fluid loss control additives from Examples 6 and 7 were added to was reduced to account for the brine present in those fluid loss control additives. The drilling fluids were each placed into a 175 mL "OFITE" HPHT Filter Press for fluid loss evaluation. In a typical experiment, pressures were adjusted to 600 and 100 psi (700 and 200 psi when the temperature is above 300°F) on the top and bottom of a sample chamber, which was filled with mud and preheated to test temperature. Drained liquid, from a filter paper with a porosity of 2.7 µm sealed on the bottom of the cylinder, was collected. The performance of each fluid loss control additive is provided below in Table 1. The acceptable performance by an additive was set to be 10 ML volume of filtrate with 30 min test.

Table 1

Fluid Loss Control Additive	Temperature (°F)	Crosslinking Agent (wt %)	Dosage (wt %)	Fluid Loss (mL)
N-DRILL HT PLUS®	250	N/A	2.0	9.5
Test Composition 1	250	0	2.0	100
Test Composition 2	250	0.006	2.0	4.3
N-DRILL HT PLUS®	300	N/A	2.0	100
Test Composition 2	300	0.006	2.0	100
Test Composition 2	300	0.006	1.0	32
Test Composition 2	300	0.006	0.5	37
Test Composition 3	300	0.7	2.0	6.3
Test Composition 3	300	0.7	1.0	16.3
Test Composition 3	300	0.7	0.5	50.0
Test Composition 3	350	0.7	2.0	40.0
Test Composition 4	350	2.7	2.0	10.2
Test Composition 5	350	1.3	2.0	7.5
Test Composition 6	350	0.3	2.0	43.5
Test Composition 7	350	1.3	2.0	6.5

[0057] In some embodiments, the present invention provides a method comprising: providing an aqueous based treatment fluid comprising a fluid loss control additive that comprises at least one polymeric micro gel; placing the aqueous based treatment fluid in a subterranean formation via a well bore penetrating the subterranean formation; allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation; allowing the filter cake to be degraded; and producing hydrocarbons from the formation.

[0058] As can be seen by Table 1, test composition 1 which represented a linear high molecular weight polymer did not show effectiveness as nearly all fluid drained out. Test composition 2 (in brine solution) was found to perform better than N-Drill HT Plus® (fluid loss of 4.3 mL compared to a fluid loss of 9.5 mL) at the same dosage at 250°F. However, when tested at 300°F, both test composition 2 and N-DRILL HT Plus® nearly failed completely during

the test at a dosage of 2 weight percent. Test composition 2 was able to reduce fluid loss to 32 mL and 37 mL at a dosage of 1 weight percent and 0.5 weight percent, respectively.

[0059] At 300°F, test composition 3 (crosslinking agent content of approximately 0.7 weight percent) controlled fluid loss around 6.3 mL, however this additive failed to meet the criteria of acceptable performance of 10 mL volume of filtrate within 30 minutes at 350°F. Test compositions 5 and 7 (crosslinking agent content of approximately 1.3 weight percent) were able to control the fluid loss around 7.5 and 6.5 mL at 350°F, respectively. However, increasing the crosslinking agent content to 2.7 weight percent (as demonstrated by composition 4) resulted in fluid loss of 10.2 mL.

[0060] As can be seen by Table 1, and further demonstrated by Figure 3, dosage has an effect on the fluid loss control. As shown in Figure 3, the fluid loss by test composition 3 decreased from 50 mL to 6.3 mL as the dosage of this additive increased from 0.5 wt percent to 2.0 weight percent. Thus, it is highly likely that the fluid loss would be further reduced at dosages higher than 2.0 weight percent, even though an optimal dosage might exist.

[0061] As can further be seen by Table 1, the filtrate volume decreased from 43.5 mL to 7.5 mL, when the crosslinking agent content of the corresponding fluid loss additive increased from 0.3 weight percent to 1.3 weight percent (See test compositions 6, 3, and 5). With a further increase of the crosslinking agent content to 2.7 weight percent (see test composition 4), the filtrate volume increased slightly to 10.2 mL. Surprisingly, synergist effect was observed when a mixture was used as the additive (See test composition 7). The mud containing this additive led to a fluid loss of only 6.5 mL, lower than those by any other non-mixed test compositions and well below the acceptable value of 10 mL. Without wishing to be limited to theory, it is believed that the synergy was probably due to the physical distribution of the polymeric micro gel particles in the mud, which enhanced the fluid loss reduction.

[0062] Therefore, the present invention is well adapted to attain the ends and advantages mentioned as well as those that are inherent therein. The particular embodiments disclosed above are illustrative only, as the present invention may be modified and practiced in different but equivalent manners apparent to those skilled in the art having the benefit of the teachings herein. Furthermore, no limitations are intended to the details of construction or design herein shown, other than as described in the claims below. It is therefore evident that the particular illustrative embodiments disclosed above may be altered or modified and all such

variations are considered within the scope and spirit of the present invention. All numbers and ranges disclosed above may vary by some amount. Whenever a numerical range with a lower limit and an upper limit is disclosed, any number and any included range falling within the range are specifically disclosed. In particular, every range of values (of the form, "from about a to about b," or, equivalently, "from approximately a to b," or, equivalently, "from approximately a-b") disclosed herein is to be understood to set forth every number and range encompassed within the broader range of values. Moreover, the indefinite articles "a" or "an", as used in the claims, are defined herein to mean one or more than one of the element that it introduces. Also, the terms in the claims have their plain, ordinary meaning unless otherwise explicitly and clearly defined by the patentee.

What is claimed is:

1. A drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel.
2. The drilling fluid of claim 1 wherein the fluid loss control additive is included in the drilling fluid in an amount up to about 5% by weight of active component of the polymeric micro gel.
3. The drilling fluid of claim 1 wherein the polymeric micro gel has a diameter in the range of about 10 nm to about 1 mm.
4. The drilling fluid of claim 1 wherein the polymeric micro gel comprises a reaction product formed by a polymerization reaction of a polymer or copolymer and a crosslinking agent.
5. The drilling fluid of claim 4 wherein the polymer or copolymer comprises at least one unit based on at least one compound selected from the group consisting of: acrylamides, vinyl alcohols, vinylpyrrolidones, vinylpyridines, acrylates, polyacrylamides, polyvinyl alcohols, polyvinylpyrrolidones, polyvinylpyridines, polyacrylates, polybutylene succinate, polybutylene succinate-co-adipate, polyhydroxybutyrate-valerate, polyhydroxybutyrate-covalerate, polycaprolactones, polyester amides, polyethylene terephthalates, sulfonated polyethylene terephthalate, polyethylene oxides, polyethylenes, polypropylenes, aliphatic aromatic copolyester, polyacrylic acids, polysaccharides (such as dextran or cellulose), chitins, chitosans, proteins, aliphatic polyesters, polylactic acids, poly(glycolides), poly(ϵ -caprolactones), poly(hydroxy ester ethers), poly(hydroxybutyrates), poly(anhydrides), polycarbonates, poly(orthoesters), poly(amino acids), poly(ethylene oxides), poly(propylene oxides), poly(phosphazenes), polyester amides, polyamides, polystyrenes, a copolymer thereof, a homopolymer thereof, a tetrapolymer thereof, and any derivative thereof.
6. The drilling fluid of claim 4 wherein the crosslinking agent comprises at least one crosslinking agent selected from the group consisting of: diethylene glycol dimethacrylate, azobisisobutyronitrile, and 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride.
7. The drilling fluid of claim 1 further comprising a bridging agent.
8. A method comprising:
providing an aqueous based treatment fluid comprising a fluid loss control additive that comprises at least one polymeric micro gel;

placing the aqueous based treatment fluid in a subterranean formation via a well bore penetrating the subterranean formation;

allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation;

allowing the filter cake to be degraded; and

producing hydrocarbons from the formation.

9. The method of claim 8 wherein the fluid loss control additive is included in the aqueous based treatment fluid in an amount up to about 5% by weight of active component of the polymeric micro gel.

10. The method of claim 8 wherein the polymeric micro gel has a diameter in the range of about 10 nm to about 1 mm.

11. The method of claim 8 wherein the polymeric micro gel comprises a reaction product formed by a polymerization reaction of a polymer or copolymer and a crosslinking agent.

12. The method of claim 11 wherein the polymer or copolymer comprises at least one unit based on at least one compound selected from the group consisting of: acrylamides, vinyl alcohols, vinylpyrrolidones, vinylpyridines, acrylates, polyacrylamides, polyvinyl alcohols, polyvinylpyrrolidones, polyvinylpyridines, polyacrylates, polybutylene succinate, polybutylene succinate-co-adipate, polyhydroxybutyrate-valerate, polyhydroxybutyrate-covalerate, polycaprolactones, polyester amides, polyethylene terephthalates, sulfonated polyethylene terephthalate, polyethylene oxides, polyethylenes, polypropylenes, aliphatic aromatic copolyester, polyacrylic acids, polysaccharides (such as dextran or cellulose), chitins, chitosans, proteins, aliphatic polyesters, polylactic acids, poly(glycolides), poly(ϵ -caprolactones), poly(hydroxy ester ethers), poly(hydroxybutyrates), poly(anhydrides), polycarbonates, poly(orthoesters), poly(amino acids), poly(ethylene oxides), poly(propylene oxides), poly(phosphazenes), polyester amides, polyamides, polystyrenes, a copolymer thereof, a homopolymer thereof, a tetrapolymer thereof, derivatives and any combinations thereof.

13. The method of claim 11 wherein the crosslinking agent comprises at least one crosslinking agent selected from the group consisting of: 2,2'-azobis-(2-methylbutyronitrile), 2,2'-azobis(isobutyramidine hydrochloride), 2,2'-azobis[2-(2-imidazolin-2-

yl)propane]dihydrochloride, 1,1'-azobis(cyclohexanecarbonitrile), 2,2'-azobis(2-methylpropionamidine) dihydrochloride, 4,4'-azobis(4-cyanovaleric acid), ammonium persulfate, hydroxymethanesulfinic acid monosodium salt dihydrate, potassium persulfate, sodium persulfate, benzoyl peroxide, 1,1-bis(*tert*-amylperoxy)cyclohexane, 1,1-bis(*tert*-butylperoxy)cyclohexane, 2,2-bis(*tert*-butylperoxy)butane, 2,4-pentanedione peroxide, 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane, 2,5-di(*tert*-butylperoxy)-2,5-dimethyl-3-hexyne; 2-butanone peroxide, cumene hydroperoxide, di-*tert*-amyl peroxide, dicumyl peroxide, lauroyl peroxide, *tert*-butyl hydroperoxide, *tert*-butyl peracetate, *tert*-butyl peroxide, *tert*-butyl peroxybenzoate, *tert*-butylperoxy-2-ethylhexyl carbonate, diethylene glycol dimethacrylate, azobisisobutyronitrile, derivatives and combinations thereof.

14. The method of claim 8 wherein the aqueous based treatment fluid further comprises a bridging agent.

15. A method comprising:

providing a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel;

circulating the drilling fluid in a subterranean formation;

allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation;

allowing the filter cake to be degraded; and

producing hydrocarbons from the formation.

16. The method of claim 15 wherein the fluid loss control additive is included in the drilling fluid in an amount up to about 5% by weight of active component of the polymeric micro gel.

17. The method of claim 15 wherein the polymeric micro gel has a diameter in the range of about 10 nm to about 1 mm.

18. The method of claim 15 wherein the polymeric micro gel comprises a reaction product formed by a polymerization reaction of a polymer or copolymer and a crosslinking agent.

19. The method of claim 18 wherein the polymer or copolymer comprises at least one unit based on at least one compound selected from the group consisting of: acrylamides, vinyl alcohols, vinylpyrrolidones, vinylpyridines, acrylates, polyacrylamides,

polyvinyl alcohols, polyvinylpyrrolidones, polyvinylpyridines, polyacrylates, polybutylene succinate, polybutylene succinate-co-adipate, polyhydroxybutyrate-valerate, polyhydroxybutyrate-covalerate, polycaprolactones, polyester amides, polyethylene terephthalates, sulfonated polyethylene terephthalate, polyethylene oxides, polyethylenes, polypropylenes, aliphatic aromatic copolyester, polyacrylic acids, polysaccharides (such as dextran or cellulose), chitins, chitosans, proteins, aliphatic polyesters, polylactic acids, poly(glycolides), poly(ϵ -caprolactones), poly(hydroxy ester ethers), poly(hydroxybutyrates), poly(anhydrides), polycarbonates, poly(orthoesters), poly(amino acids), poly(ethylene oxides), poly(propylene oxides), poly(phosphazenes), polyester amides, polyamides, polystyrenes, a copolymer thereof, a homopolymer thereof, a tetrapolymer thereof, derivatives and combinations thereof.

20. The method of claim 18 wherein the crosslinking agent comprises at least one crosslinking agent selected from the group consisting of: 2,2'-azobis-(2-methylbutyronitrile), 2,2'-azobis(isobutyramidine hydrochloride), 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride, 1,1'-azobis(cyclohexanecarbonitrile), 2,2'-azobis(2-methylpropionamidine) dihydrochloride, 4,4'-azobis(4-cyanovaleric acid), ammonium persulfate, hydroxymethanesulfinic acid monosodium salt dihydrate, potassium persulfate, sodium persulfate, benzoyl peroxide, 1,1-bis(*tert*-amylperoxy)cyclohexane, 1,1-bis(*tert*-butylperoxy)cyclohexane, 2,2-bis(*tert*-butylperoxy)butane, 2,4-pentanedione peroxide, 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane, 2,5-di(*tert*-butylperoxy)-2,5-dimethyl-3-hexyne; 2-butanone peroxide, cumene hydroperoxide, di-*tert*-amyl peroxide, dicumyl peroxide, lauroyl peroxide, *tert*-butyl hydroperoxide, *tert*-butyl peracetate, *tert*-butyl peroxide, *tert*-butyl peroxybenzoate, *tert*-butylperoxy-2-ethylhexyl carbonate, diethylene glycol dimethacrylate, azobisisobutyronitrile, derivatives and combinations thereof.

21. The method of claim 15 wherein the drilling fluid further comprises a bridging agent.

22. A method comprising:

providing an inside screen pill comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel, and

placing the inside screen pill in a subterranean formation so that the inside screen pill is located between a gravel pack screen and an area of the subterranean formation.

23. The method of claim 22 wherein the fluid loss control additive is included in the inside screen pill in an amount of about 1% to about 10% by weight of active component of the polymeric micro gel.

24. The method of claim 22 wherein the polymeric micro gel has a diameter in the range of about 10 nm to about 1 mm.

25. The method of claim 22 wherein the polymeric micro gel comprises a reaction product formed by a polymerization reaction of a polymer or copolymer and a crosslinking agent.

26. The method of claim 25 wherein the crosslinking agent comprises at least one crosslinking agent selected from the group consisting of: 2,2'-azobis-(2-methylbutyronitrile), 2,2'-azobis(isobutyramidine hydrochloride), 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride, 1,1'-azobis(cyclohexanecarbonitrile), 2,2'-azobis(2-methylpropionamidine) dihydrochloride, 4,4'-azobis(4-cyanovaleric acid), ammonium persulfate, hydroxymethanesulfonic acid monosodium salt dihydrate, potassium persulfate, sodium persulfate, benzoyl peroxide, 1,1-bis(*tert*-amylperoxy)cyclohexane, 1,1-bis(*tert*-butylperoxy)cyclohexane, 2,2-bis(*tert*-butylperoxy)butane, 2,4-pentanedione peroxide, 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane, 2,5-di(*tert*-butylperoxy)-2,5-dimethyl-3-hexyne; 2-butanone peroxide, cumene hydroperoxide, di-*tert*-amyl peroxide, dicumyl peroxide, lauroyl peroxide, *tert*-butyl hydroperoxide, *tert*-butyl peracetate, *tert*-butyl peroxide, *tert*-butyl peroxybenzoate, *tert*-butylperoxy-2-ethylhexyl carbonate, diethylene glycol dimethacrylate, azobisisobutyronitrile, derivatives and combinations thereof.

27. The method of claim 25 wherein the polymer or copolymer comprises at least one unit based on at least one compound selected from the group consisting of: acrylamides, vinyl alcohols, vinylpyrrolidones, vinylpyridines, acrylates, polyacrylamides, polyvinyl alcohols, polyvinylpyrrolidones, polyvinylpyridines, polyacrylates, polybutylene succinate, polybutylene succinate-co-adipate, polyhydroxybutyrate-valerate, polyhydroxybutyrate-covalerate, polycaprolactones, polyester amides, polyethylene terephthalates, sulfonated polyethylene terephthalate, polyethylene oxides, polyethylenes, polypropylenes, aliphatic aromatic copolyester, polyacrylic acids, polysaccharides (such as dextran or cellulose), chitins, chitosans, proteins, aliphatic polyesters, polylactic acids, poly(glycolides), poly(ϵ -caprolactones), poly(hydroxy ester ethers), poly(hydroxybutyrates),

poly(anhydrides), polycarbonates, poly(orthoesters), poly(amino acids), poly(ethylene oxides), poly(propylene oxides), poly(phosphazenes), polyester amides, polyamides, polystyrenes, a copolymer thereof, a homopolymer thereof, a tetrapolymer thereof, and any derivative thereof.

ABSTRACT

Methods and fluids are provided that include, but are not limited to, a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel and a method comprising: providing an aqueous based treatment fluid comprising a fluid loss control additive that comprises at least one polymeric micro gel; placing the aqueous based treatment fluid in a subterranean formation via a well bore penetrating the subterranean formation; allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation; allowing the filter cake to be degraded; and producing hydrocarbons from the formation. Additional methods are also provided.

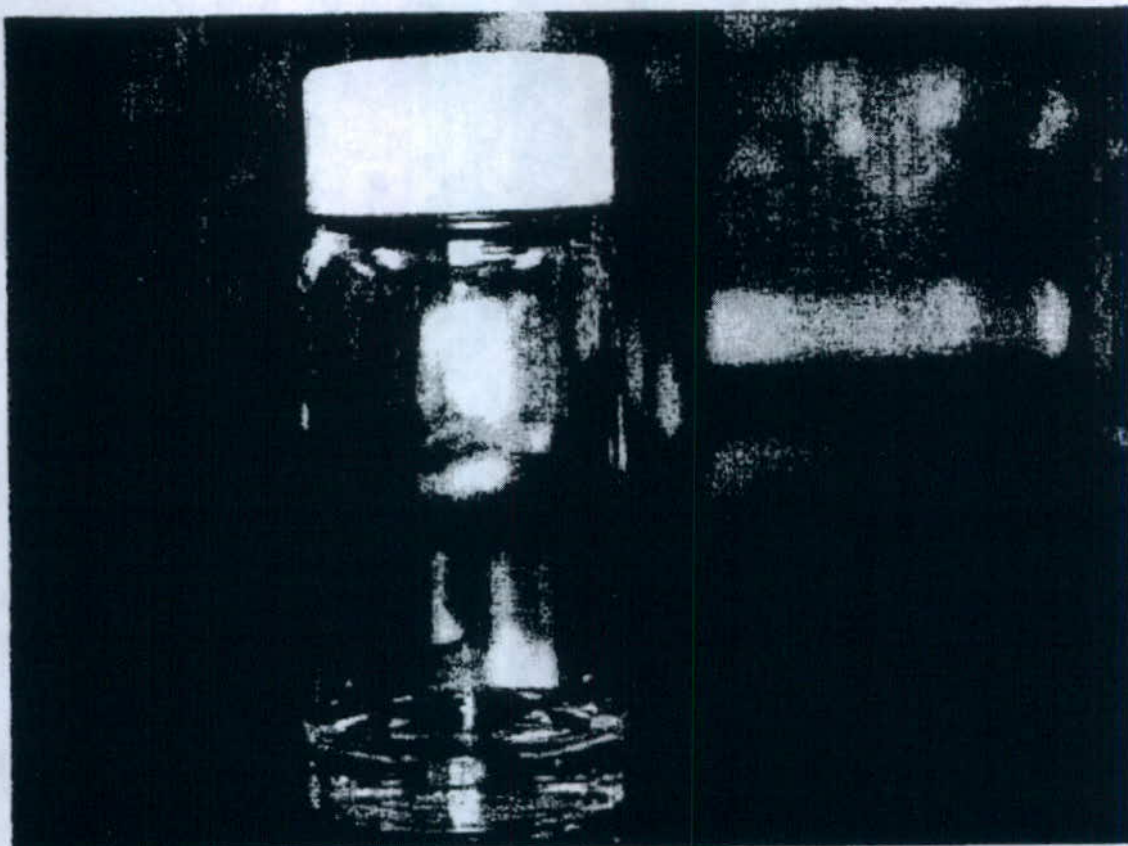


Figure 1

1/2

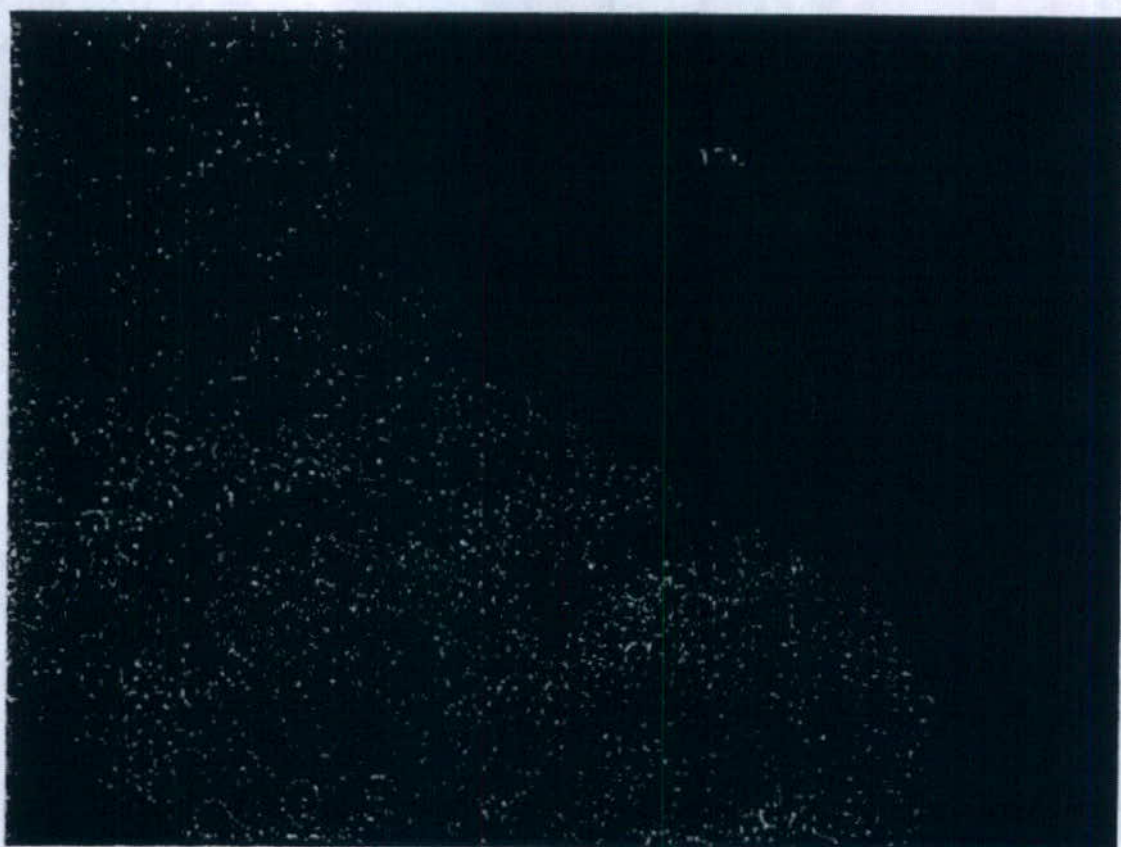


Figure 2

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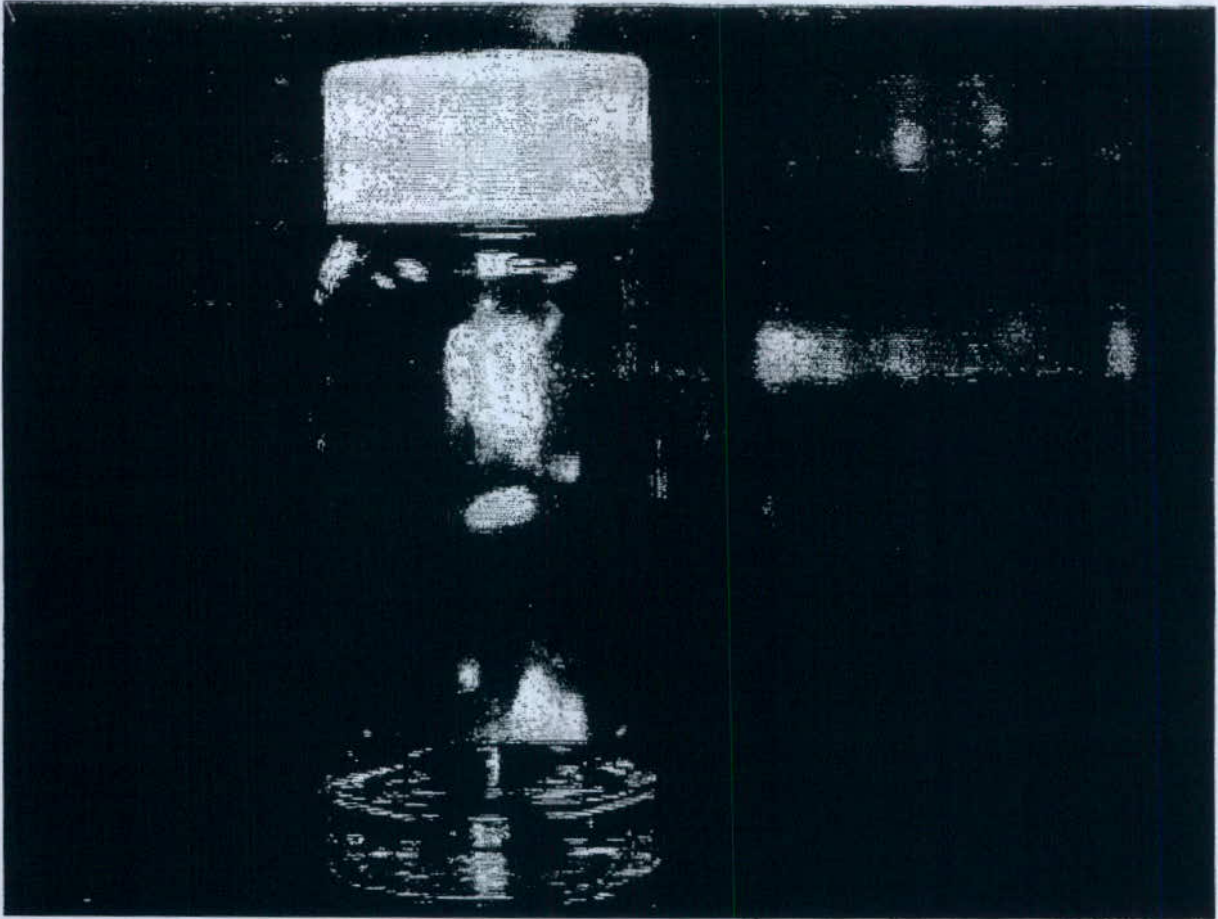


Figure 1



Figure 2

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
7 October 2010 (07.10.2010)



(10) International Publication Number
WO 2010/112866 A1

(51) International Patent Classification:
C09K 8/512 (2006.01)

(21) International Application Number:
PCT/GB2010/000657

(22) International Filing Date:
1 April 2010 (01.04.2010)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
12/418,323 3 April 2009 (03.04.2009) US

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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

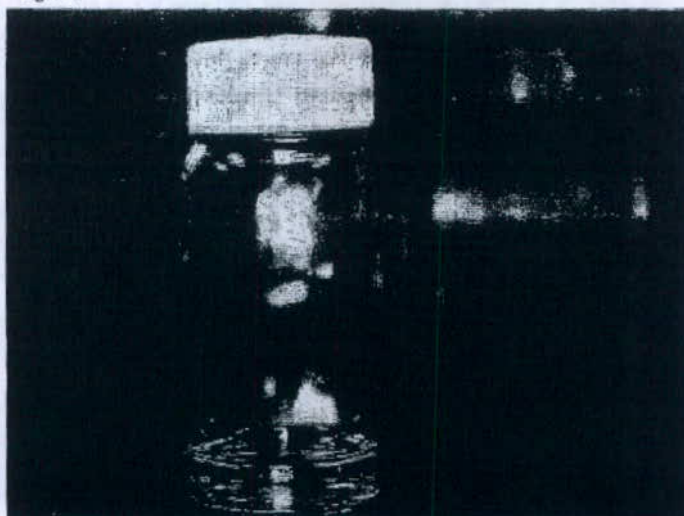
(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

- with international search report (Art. 21(3))
- before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))

(54) Title: METHODS OF USING FLUID LOSS ADDITIVES COMPRISING MICRO GELS

Figure 1



(57) Abstract: Methods and fluids are provided that include, but are not limited to, a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel and a method comprising: providing an aqueous based treatment fluid comprising a fluid loss control additive that comprises at least one polymeric micro gel; placing the aqueous based treatment fluid in a subterranean formation via a well bore penetrating the subterranean formation; allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation; allowing the filter cake to be degraded; and producing hydrocarbons from the formation. Additional methods are also provided.

97

METHODS OF USING FLUID LOSS ADDITIVES COMPRISING MICRO GELS

BACKGROUND

[0001] The present invention relates to methods of using fluid loss control additives. More specifically, at least in some embodiments, the present invention relates to the use of fluid loss control additives in drilling and servicing fluids that comprise polymeric micro gels in subterranean operations.

[0002] When well bores are drilled into producing formations, drilling fluids are utilized which will minimize damage to the permeability of the formations and their ability to produce hydrocarbons. Servicing fluids are utilized when completion operations are conducted in producing formations and when conducting work-over operations in the formations. The drilling and servicing fluids deposit a layer of particles known as "filter cake" on the walls of the well bores within the producing formations. The filter cake is believed to help prevent the drilling and servicing fluids from being lost into the formation and prevents solids from entering the porosities of the rock. Following completion and prior to initiating production, the filter cake is usually degraded or allowed to degrade to allow product to flow into the well bore for production. Degrading the filter cake is important to retain well bore connectivity and the natural permeability of the reservoir rock. If not degraded or allowed to degrade, the filter cake could present an impediment to production, *inter alia*, by altering the permeability of the reservoir. Once the permeability of the reservoir has been diminished, it is seldom able to restore it to its original condition. These should be distinguished from the function of additives (sometimes termed in the art "relative permeability modifiers" or "RPMs") that are often used in conformance or fracturing fluids to permanently seal water influxes to hydrocarbon reservoir areas surrounding a well bore.

[0003] Drilling and servicing fluids (such as drill-in fluids) may comprise fluid loss control additives to further assist in preventing the drilling and servicing fluids from being lost into the formations. Drilling fluids are any of a number of fluids and mixtures of fluids and solids (as solid suspensions, mixtures and emulsions of liquids, gases and solids) used in operations to drill boreholes into the earth. Classifications of drilling fluids has been attempted in many ways, often producing more confusion than insight. One classification scheme, given here, is based only on the mud composition by singling out the component that clearly defines the function and performance of the fluid: (1) aqueous-based, (2) oil-based and (3) gaseous

(pneumatic). Each category has a variety of subcategories that overlap each other considerably. Ideally, a drilling fluid is non-damaging to the formation, meaning that the fluid does not leave behind particulates, fines, etc. that negatively impact the permeability of the formation.

[0004] A drill-in fluid is a fluid designed for drilling through the reservoir section of a well bore in a subterranean formation. The reasons for using a specially designed fluid include, but are not necessarily limited to: (1) to drill the reservoir zone successfully, often a long, horizontal drain hole; (2) to minimize damage to and maximize production of exposed zones; and (3) to facilitate the well completion needed, which may include complicated procedures. A drill-in fluid often resembles a completion fluid in that it may comprise a brine, possibly bridging agents, and/or polymers.

[0005] The term "drilling fluid" as used herein refers generically to both drilling fluids and drill-in fluids unless otherwise specified.

[0006] Other types of treatment fluids that can utilize fluid loss control materials include, but are not limited to, pills (such as inside screen pills), which are fluids with a relatively small quantity (*e.g.*, less than 200 bbl) of a special blend of drilling fluid to accomplish a specific task that the regular drilling fluid cannot perform. Examples include high-viscosity pills to help lift cuttings out of a vertical well bore, freshwater pills to dissolve encroaching salt formations, pipe-freeing pills to destroy filter cake and relieve differential sticking forces, and lost circulation material pills to plug a thief zone. Another example is a screen pill that may be useful in conjunction with a gravel pack operation.

[0007] Examples of conventional fluid loss control additives for water-based treatment fluids include nonionic water soluble polymers, such as starches, derivatized starches, gums, derivatized gums, and celluloses. Fluid loss additives that include starches often vary in the ratio of amylose to amylopectin content, and may or may not be modified with a crosslinking agent such as epichlorohydrin. Also, natural starches may not be uniform in terms of quality and effectiveness. These cross-linked starches often do not have thermal stability at temperatures up to about 250 °F, and at temperatures above 250 °F, they can only effectively be used by increased loading the treatment fluid with the cross-linked starch, constantly replenishing the treatment fluid with the cross-linked starch, or using an oxygen scavenger in conjunction with the cross-linked starch. At temperatures above 300 °F, even by the use of the above mentioned measures, cross-linked starches may not be effective fluid loss control additives.

[0008] Conventional linear synthetic polymers are also utilized, but oftentimes, they require another additive, such as a clay, to be able to effectively function as fluid loss control additives. However, the use of clay can be problematic in drill-in fluids, as removing the clay from the subterranean formation can be difficult because it infiltrates into pores of the subterranean formation. Furthermore, the addition of clay to a treatment fluid dramatically increases the viscosity of the fluid, which can cause drilling to be completed at a reduced rate. Another issue is that some synthetic polymers cannot be used successfully in conjunction with some brines, for instance, divalent brines. This brine incompatibility is thought to prevent the synthetic polymer from associating with the clay to forming bridging colloids, which are desirable because they provide a degree of fluid loss, *e.g.*, in a drill-in fluid.

SUMMARY

[0009] The present invention relates to methods of using fluid loss control additives. More specifically, at least in some embodiments, the present invention relates to the use of fluid loss control additives in drilling and servicing fluids that comprise polymeric micro gels in subterranean operations.

[0010] In one embodiment, the present invention provides a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel.

[0011] In another embodiment, the present invention provides a method comprising: providing an aqueous based treatment fluid comprising a fluid loss control additive that comprises at least one polymeric micro gel; placing the aqueous based treatment fluid in a subterranean formation via a well bore penetrating the subterranean formation; allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation; allowing the filter cake to be degraded; and producing hydrocarbons from the formation.

[0012] In another embodiment, the present invention provides a method comprising: providing a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel; circulating the drilling fluid in a subterranean formation; allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation; allowing the filter cake to be degraded; and producing hydrocarbons from the formation.

[0013] In another embodiment, the present invention provides a method comprising: providing an inside screen pill comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel, and placing the inside screen pill in a subterranean formation so that the inside screen pill is located between a gravel pack screen and an area of the subterranean formation.

[0014] The features and advantages of the present invention will be readily apparent to those skilled in the art. While numerous changes may be made by those skilled in the art, such changes are within the spirit of the invention.

BRIEF DESCRIPTION OF THE DRAWINGS

[0015] These drawings illustrate certain aspects of some of the embodiments of the present invention, and should not be used to limit or define the invention.

[0016] Figure 1 is a photograph of a transparent solution of a micro gel dispersion after dissolving in water.

[0017] Figure 2 is a micrograph of the micro gel dispersion of Figure 1.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0018] The present invention relates to methods of using fluid loss control additives. More specifically, at least in some embodiments, the present invention relates to the use of fluid loss control additives in drilling and servicing fluids that comprise polymeric micro gels in subterranean operations.

[0019] There may be several potential advantages to the methods and compositions of the present invention, only some of which are alluded to herein. The present application provides drilling fluids, preferably for use as a drilling, drill-in, completion fluid or pills, which quickly forms a thin, degradable filter cake. Again, for the sake of clarity, the term "drilling fluid" as used herein refers generically to both drilling fluids and drill-in fluids unless otherwise specified. The drilling fluids of the present invention are less invasive to the formation yet provide lubricity, effective fluid loss control, and good filter cake sealing and plastering characteristics. The resulting filter cake can be easily lifted off and readily removed or dissolved to help ensure low skin and residual damage. Moreover, the fluids are stable for prolonged periods of time at high temperatures. In particular, the drilling fluids of the present invention are believed to have enhanced performance over conventional fluids that comprise starch derivatives, for instance, because they can be used at effective static bottom hole temperatures up to at least about 400 °F or more while maintaining effective fluid loss control, thus minimizing formation damage. In doing so, a vast range of applications is open for technological advancement and new product revenue. It is believed that in equal concentrations, the drilling fluids of the present invention that comprise fluid loss additives comprising polymeric micro gels described herein give an increased performance (e.g., an order of magnitude increase) in the amount of fluid loss over starch-based fluid loss materials.

[0020] Another potential advantage may be that the drilling fluids of the present invention may be used without the addition of clay, which allows for easier clean up, minimal formation damage, faster drilling times, and the avoidance of problems associated with the increased viscosity of fluids when clay is added.

[0021] In some embodiments, the present invention provides an aqueous-based drilling fluid that comprises a fluid loss control additive of the present invention that comprises at least one polymeric micro gel. In other embodiments, the present invention provides drilling fluids that comprise an aqueous base fluid (e.g., a brine or fresh water), and one or more fluid

loss control additives that comprise at least one polymeric micro gel. In practical usage, with respect to building a filter cake that will easily degrade, it may be beneficial to include bridging agents in the drilling fluid that will become incorporated into the filter cake. Without the bridging agents, the polymeric micro gels may form an effective filter cake, but that filter cake may be more difficult to remove. Other components of the drilling fluid may comprise weighting agents, clays, polymers, as well as other additives common to aqueous-based drilling fluids including, but not necessarily limited to, lubricants, corrosion inhibitors, other inhibitors, and oxygen scavengers.

[0022] In some embodiments, the fluid loss control additive may be included in the drilling fluid in an amount of about 5% or below by weight of active component of the polymeric micro gel.

[0023] The base fluid of the drilling fluids of the present invention may be any suitable aqueous-based fluid including, but not necessarily limited to, brines (*i.e.*, water comprising a salt). In some instances, brines having a density of at least about 9 lb/gal, referred to as "high density brines," may be suitable. The brines may contain substantially any suitable salts, including, but not necessarily limited to, salts based on metals, such as calcium, magnesium, sodium, potassium, cesium, zinc, aluminum, and lithium. Salts of calcium and zinc are preferred. The salts may contain substantially any anions, with preferred anions being less expensive anions including, but not necessarily limited to chlorides, bromides, formates, acetates, and nitrates. Generally, the base fluid is present in a drilling fluid of the present invention in an amount in the range of from about 35% to about 97% by weight thereof, more preferably from about 70% to about 95% or more.

[0024] Preferably, when used, the bridging agents are either self-degrading or degradable in a suitable clean-up solution (*e.g.*, a mutual solvent, water, an acid solution, etc.). Examples of bridging agents suitable for use in the methods of the current invention include, but are not necessarily limited to, magnesium citrate, calcium citrate, calcium succinate, calcium maleate, calcium tartrate, magnesium tartrate, bismuth citrate, calcium carbonate, sodium chloride and other salts, and the hydrates thereof. Examples of degradable bridging agents may include, but are not necessarily limited to, bridging agents comprising degradable materials such as degradable polymers. A polymer is considered to be "degradable" herein if the degradation is due to, *inter alia*, chemical and/or radical process such as hydrolysis, oxidation, enzymatic

degradation, or UV radiation. Suitable examples of degradable polymers that may be used in accordance with the present invention include, but are not necessarily limited to, those described in the publication of *Advances in Polymer Science*, Vol. 157 entitled "Degradable Aliphatic Polyesters" edited by A. C. Albertsson, the disclosure of which is hereby incorporated by reference. Specific examples of suitable polymers include, but are not necessarily limited to, polysaccharides such as dextrans or celluloses; chitins; chitosans; proteins; orthoesters; aliphatic polyesters; poly(lactides); poly(glycolides); poly(ϵ -caprolactones); poly(hydroxybutyrates); poly(anhydrides); aliphatic polycarbonates; poly(orthoesters); poly(amino acids); poly(ethylene oxides); and polyphosphazenes. Combinations and derivatives of these are suitable as well.

[0025] When choosing a particular bridging agent to use, one should be aware of the performance of that bridging agent at the temperature range of the application. The bridging agents utilized may be generally present in the drilling fluid compositions in an amount in the range of from about 1% to about 40% by weight thereof, more preferably from about 5% to about 25%. Generally, the bridging agents may have a particle size in the range of from about 1 micron to about 600 microns. Preferably, the bridging particle size is in the range of from about 1 to about 200 microns but may vary from formation to formation. The particle size used is determined by the pore throat size of the formation.

[0026] The term "polymeric micro gel" as used herein refers to a gelled particle comprising a cross-linked polymer (*e.g.*, water-soluble and water-swelling) network. In some embodiments, the fluid loss control additive may be delivered in a continuous medium, *e.g.*, in a dispersion form. In other embodiments, the polymeric micro gels may be used without any continuous medium, *e.g.*, in dry form. One of the many useful features of certain embodiments of the polymeric micro gels of the present invention is that they are believed to swell or shrink in response to an external stimuli, *e.g.*, pH and temperature. In some embodiments, some of the polymeric micro gels may be considered to be super absorbent in that they can contain over 99% water. This may be advantageous in some applications wherein water absorbance is important.

[0027] When comprising a substantial portion of the fluid loss control additive in a drilling fluid of the present invention, the polymeric micro gels optionally may be dispersed in a continuous medium before addition to the fluid. In some embodiments, the fluid loss control additive may comprise a continuous medium. Examples of suitable continuous mediums may include, but are not necessarily limited to, aqueous-based fluids, alcohols, glycerin, glycols,

polyglycol amines, polyols, and any derivative thereof. Additionally, in some embodiments, the continuous medium may comprise a fluid selected from the group consisting of methanol, ethanol, *n*-propanol, isopropanol, *n*-butanol, *sec*-butanol, isobutanol, *t*-butanol, a mixture of methanol, ethanol, *n*-propanol, isopropanol, *n*-butanol, *sec*-butanol, isobutanol, or *t*-butanol and water, a mixture of ammonium sulfate, sodium sulfate, or potassium sulfate and water, a mixture of sodium chloride, potassium chloride, or calcium chloride and water, and combinations thereof. Optionally, the continuous medium comprises a fluid selected from the group consisting of ethanol, a mixture of *t*-butanol and water, and a mixture of ammonium sulfate and water. Mixtures of these may be suitable as well. Examples of suitable aqueous-based fluids may include, but are not necessarily limited to, fresh water, sea water, salt water, and brines (e.g., saturated salt waters). Examples of suitable brines may include, but are not necessarily limited to, heavy brines, monovalent brines, divalent brines, and trivalent brines that comprise a soluble salts like sodium chloride, calcium chloride, calcium bromide, zinc bromide, potassium carbonate, sodium formate, potassium formate, cesium formate, sodium acetate, potassium acetate, calcium acetate, ammonium acetate, ammonium chloride, ammonium bromide, sodium nitrate, potassium nitrate, ammonium nitrate, ammonium sulfate, calcium nitrate, sodium carbonate, potassium carbonate, any combination thereof, and any derivative thereof. Examples of suitable alcohols may include, but are not necessarily limited to, methanol, ethanol, propanol, iso-propanol, butanol, tert-butanol, and the like. Examples of suitable glycols may include, but are not necessarily limited to, polyglycols, propylene glycol, ethylene glycol, and the like. In some embodiments, the continuous medium may be present in any suitable range including ratios of continuous medium to polymeric micro gel of about 30:70, about 20:80, about 10:90, about 5:95, and about 0:100 by weight.

[0028] In some embodiments, the polymeric micro gels may comprise a reaction product formed by a suitable polymerization reaction of a polymer (or monomer) and a crosslinking agent. In one embodiment of a method of forming the micro gels of the present invention, the polymerization is a dispersion polymerization in a continuous medium that is substantially inert toward chain transfer reactions. Crosslinked polymer micro gel particles are formed, which are believed to be insoluble or at most swellable in the continuous medium. While not wishing to be limited to any particular theory, it is believed that in some embodiments, the crosslinking agent may act as an initiator forming chain branches of the polymer or

monomer, which in turn may react with one another to form a polymeric micro gel. Other suitable initiators may include those known to those skilled in the art such as photoinitiators, thermal initiators, and combinations thereof.

[0029] In some embodiments, the polymer (or monomer) may comprise any suitable polymer or monomer that can form a polymeric micro gel when crosslinked. Suitable polymers and monomers may include, but are not necessarily limited to, those that comprise units based on acrylamides, vinyl alcohols, vinylpyrrolidones, vinylpyridines, acrylates, polyacrylamides, polyvinyl alcohols, polyvinylpyrrolidones, polyvinylpyridines, polyacrylates, polybutylene succinate, polybutylene succinate-co-adipate, polyhydroxybutyrate-valerate, polyhydroxybutyrate-covalerate, polycaprolactones, polyester amides, polyethylene terephthalates, sulfonated polyethylene terephthalate, polyethylene oxides, polyethylenes, polypropylenes, aliphatic aromatic copolyester, polyacrylic acids, polysaccharides (such as dextran or cellulose), chitins, chitósans, proteins, aliphatic polyesters, polylactic acids, poly(glycolides), poly(ϵ -caprolactones), poly(hydroxy ester ethers), poly(hydroxybutyrates), poly(anhydrides), polycarbonates, poly(orthoesters), poly(amino acids), poly(ethylene oxides), poly(propylene oxides), poly(phosphazenes), polyester amides, polyamides, polystyrenes, any derivative thereof, any copolymer, homopolymer, or terpolymer, or any blend thereof. In some embodiments, the monomer may include an unsaturated group, such as a monomer including a vinyl group. Exemplary vinyl-containing monomers may be described by the formula $C(R_1)(R_2)=C(R_3)(R_4)$, wherein R_1 , R_2 , R_3 and R_4 are segments rendering the solubility or swellability of this monomer in the common solvent. Optionally, R_1 , R_2 , R_3 and R_4 can each be independently selected from, but not limited to, hydrogen, methyl, ethyl, $CONH_2$, $CONHCH_3$, $CON(CH_3)_2$, CH_2SO_3H , CH_2SO_3Na and $COONa$. In a further option, the monomer comprises a compound selected from the group consisting of hydroxyethyl acrylate, acrylamide and hydroxyethyl methacrylate. Examples of suitable copolymers that may be used with these may include inorganic and organic polymers. Some of these polymers and monomers may be more suited for less high temperature applications than others; however, crosslinking may increase their thermal stability. In some embodiments the polymer or monomer may comprise a water soluble polymer or monomer. In some embodiments the polymer or monomer may comprise a block copolymer with a portion that is water soluble and a portion that is water insoluble.

[0030] In some embodiments, the crosslinking agent may comprise any crosslinking agent that can react with a polymer or monomer to form a polymeric micro gel. In some embodiments, the crosslinking agent may comprise a covalent crosslinking agent. Examples of suitable crosslinking agents may include crosslinking agents that form a radical. Suitable crosslinking agents may include, but are not limited to, 2,2'-azobis-(2-methylbutyronitrile), 2,2'-azobis(isobutyramidine hydrochloride), 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride, 1,1'-azobis(cyclohexanecarbonitrile), 2,2'-azobis(2-methylpropionamidine) dihydrochloride, 4,4'-azobis(4-cyanovaleric acid), ammonium persulfate, hydroxymethanesulfinic acid monosodium salt dihydrate, potassium persulfate, sodium persulfate, benzoyl peroxide, 1,1-bis(*tert*-amylperoxy)cyclohexane, 1,1-bis(*tert*-butylperoxy)cyclohexane, 2,2-bis(*tert*-butylperoxy)butane, 2,4-pentanedione peroxide, 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane, 2,5-di(*tert*-butylperoxy)-2,5-dimethyl-3-hexyne; 2-butanone peroxide, cumene hydroperoxide, di-*tert*-amyl peroxide, dicumyl peroxide, lauroyl peroxide, *tert*-butyl hydroperoxide, *tert*-butyl peracetate, *tert*-butyl peroxide, *tert*-butyl peroxybenzoate, *tert*-butylperoxy-2-ethylhexyl carbonate, diethylene glycol dimethacrylate, azobisisobutyronitrile, and combinations thereof. A person of ordinary skill in the art would be able to select an appropriate crosslinking agent based upon the polymer or monomer to be crosslinked.

[0031] In some embodiments, the amount of crosslinking agent used in conjunction with the polymer or monomer may be any amount necessary to create the desired degree of crosslinking, and other characteristics. In some embodiments, the ratio of crosslinking agent to monomer or polymer used to form the polymeric micro gel may be in the range of from about 100:1 to about 5,000:1 by weight.

[0032] In one embodiment of a method of forming the micro gels of the present invention, the polymerization is a dispersion polymerization in a continuous medium such as a continuous medium comprising *t*-butyl alcohol/water and ammonium sulfate. Both the monomer (such as, but not limited to, acrylamide) and the crosslinking agent (such as, but not limited to, *N,N'*-ethylene-bis-acrylamide) are substantially soluble or at least swellable in the continuous medium. The resultant micro gels are not soluble in the continuous medium. The continuous medium is believed to be relatively inert (or at least not so reactive as to disrupt micro gels from

forming) toward chain transfer reactions by propagating radicals. By removing the continuous medium using a suitable technique, the micro gels may be dried if desired.

[0033] Optionally, a colloidal stabilizer may also be included in the continuous medium during polymerization. One example of a colloidal stabilizer is amphiphilic, such as an amphiphilic colloidal stabilizer comprising a stabilizer selected from the group consisting of poly(vinyl pyrrolidone) (PVP), polydiallyldimethylammonium chloride (poly-DADMAC), and combinations thereof.

[0034] In one embodiment of a method of forming the micro gels of the present invention, the polymerization is a dispersion polymerization of water-soluble or swellable vinyl monomer(s) (as represented by $C(R1)(R2)=C(R3)(R4)$, where R1, R2, R3 and R4 are segments rendering the solubility or swellability of this monomer to water and the polymerization medium) in the presence of radically polymerizable crosslinking agent (such as, but not limited to, $C(R5)(R6)=C(R7)-R8-C(R9)=C(R10)(R11)$, where R5 - R11 are groups rendering solubility and swellability of this crosslinking agent to water and the polymerization medium) in a medium (such as, but not limited to, t-butanol/water and ammonium sulfate solution in water), to which the chain transfer of propagating radical is avoided or suppressed. By taking advantage of the poor stability of the resultant dispersion at high polymer content (e.g., 13 wt %), in the absence of any crosslinking agent, the result may be one chunk of macro gel.

[0035] The size range for these polymeric micro gels may range from about 10 nm to about 1 mm in diameter. A suitable size range may be 10 microns to about 500 microns. In a given composition, a range of disparate sized polymeric micro gels may be used. One of ordinary skill in the art would be able to select an appropriate size of the polymeric micro gels based upon the characteristics of the subterranean formation in which the polymeric micro gels would be used, the temperature of the subterranean formation, the degree of stability required of the polymeric micro gels, and the costs. Also, the shapes of the polymeric micro gels may vary, and may depend on several factors including, but not necessarily limited to, temperature, the continuous medium used, and the degree of crosslinking. The polymeric micro gels can be any shape, for example, at least partially spherical. Depending on the method in which the polymeric micro gels are created, the shape of the polymeric micro gels can vary. One of ordinary skill in the art would be able to select an appropriate shape of the polymeric micro gels based upon the characteristics of the subterranean formation in which the polymeric micro gels would be used,

the temperature of the subterranean formation, the degree of stability required of the polymeric micro gels, and the costs. No particular shape is critical for use in the present invention.

[0036] In some embodiments, the fluid loss control additives of the present invention may not be delivered with a continuous medium. These may be referred to as the "dry" embodiments. For example, in some embodiments, after the polymeric micro gels are polymerized, the polymeric micro gels may be removed from the continuous medium and then dried using a suitable technique to form the powdered polymeric micro gels.

[0037] In some embodiments, the present invention comprises an aqueous based drill-in fluid comprising a fluid loss control additive of the present invention that comprises at least one polymeric micro gel. In some embodiments, a fluid loss control additive may be included in the drill-in fluid in an amount of about 5% or below by weight of active component of the polymeric micro gel. Other components of the drill-in fluids may comprise bridging agents and other commonly used additives.

[0038] In some embodiments, the present invention comprises an inside screen pill comprising a water-based fluid and a fluid loss control additive comprising at least one polymeric micro gel. In some embodiments, fluid loss control additive may be included in the inside screen pill in an amount of about 1 to about 10% by weight of active component of the polymeric micro gel.

[0039] In some embodiments, the present invention provides a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel.

[0040] In some embodiments, the present invention provides a method comprising: providing an aqueous based treatment fluid comprising a fluid loss control additive that comprises at least one polymeric micro gel; and placing the aqueous based treatment fluid in a subterranean formation via a well bore penetrating the subterranean formation.

[0041] In some embodiments, the present invention provides a method comprising: providing a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel; and circulating the drilling fluid in a subterranean formation.

[0042] In some embodiments, the present invention provides a method comprising: providing an inside screen pill comprising an aqueous base fluid and a fluid loss

control additive that comprises at least one polymeric micro gel, and placing the inside screen pill in a subterranean formation so that the inside screen pill is located between a gravel pack screen and an area of the subterranean formation.

[0043] To facilitate a better understanding of the present invention, the following examples of certain aspects of some embodiments are given. In no way should the following examples be read to limit, or define, the entire scope of the invention.

EXAMPLES

Example 1

[0044] Polymeric micro gel particles were prepared in accordance with the following procedure. A mixture of 95.0 g of ethanol, 20.0 g of acrylamide, 0.50 g of poly(vinyl pyrrolidone), and 0.50 g of diethylene glycol dimethacrylate was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles with sizes from 10 μm to 100 μm (in water) was obtained with solids content of approximately 17% by weight. Figure 1 is a photograph of the transparent micro gel dispersion formed in this example. Figure 2 is a micrograph of the micro gel dispersion formed in this example. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 2

[0045] Polymeric micro gel particles were prepared in accordance with the following procedure. A mixture of 95.0 g of a mixture of t-butanol and water (90% t-butanol by weight), 15.0 g of acrylamide, 0.20 g of N,N'-ethylene-bis-acrylamide, and 0.50 g of poly(vinyl pyrrolidone) was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a

dispersion of particles with sizes from 10 μm to 300 μm was obtained with a solids content of approximately 13% by weight.

Example 3

[0046] Polymeric micro gel particles were prepared in accordance with the following procedure. A mixture of 75.5 g of a 40% solution of ammonium sulfate in water, 7.5 g of acrylamide, 12.0 g of a 20% solution of polydiallyldimethylammonium chloride in water, 6.7 μL of poly(ethylene glycol) diacrylate, and 20.0 g of de-ionized water was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 35°C. A 5.0 g solution of 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride in water (0.01% 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride by weight) was then injected into the mixture. The mixture was then kept at 35°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles with a solid content of approximately 9% by weight was obtained. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 4

[0047] Polymeric micro gel particles were prepared in accordance with the following procedure. A mixture of 90.0 g of a mixture of t-butanol and water (90% t-butanol by weight), 15.0 g of acrylamide, 0.50 g of poly(vinyl pyrrolidone), was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. After approximately 20 minutes a solution of 0.10 g of N,N'-ethylene-bis-acrylamide in 5.0 g of a mixture of t-butanol and water (90% t-butanol by weight) was injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles with sizes from 10 μm to 300 μm was obtained with a solid content of approximately 13% by weight. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then

decanted and sediments were collected. The sediments were then dried under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 5

[0048] Polymeric micro gel particles were prepared in accordance with the following procedure. A mixture of 75.5 g of a 40% solution of ammonium sulfate in water, 7.5 g of acrylamide, 12.0 g of a 20% solution of polydiallyldimethylammonium chloride in water, 67 µL of poly(ethylene glycol) diacrylate, and 20.0 g of de-ionized water was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 35°C. A 5.0 g solution of 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride in water (0.01% 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride by weight) was then injected into the mixture. The mixture was then kept at 35°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles was obtained with a solid content of approximately 9% by weight. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 6

[0049] Test composition 1 was prepared in accordance with the following procedure. A mixture of 75.5 g of a 40% solution of ammonium sulfate in water, 7.5 g of acrylamide, 12.0 g of a 20% solution of polydiallyldimethylammonium chloride in water, and 20.0 g of de-ionized water was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 35°C. A 5.0 g solution of 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride in water (0.01% 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride by weight) was then injected into the mixture. The mixture was then kept at 35°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles was obtained with a solid content of approximately 9% by weight.

Example 7

[0050] Test composition 2 was prepared in accordance with the following procedure. A mixture of 75.5 g of a 40% solution of ammonium sulfate in water, 7.5 g of

acrylamide, 12.0 g of a 20% solution of polydiallyldimethylammonium chloride in water, 6.7 μ L of poly(ethylene glycol) diacrylate, and 20.0 g of de-ionized water was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 35°C. A 5.0 g solution of 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride in water (0.01% 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride by weight) was then injected into the mixture. The mixture was then kept at 35°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles with a solid content of approximately 9% by weight was obtained.

Example 8

[0051] Test composition 3 was prepared in accordance with the following procedure. A mixture of 90.0 g of a mixture of t-butanol and water (90% t-butanol by weight), 15.0 g of acrylamide, 0.50 g of poly(vinyl pyrrolidone), was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. After approximately 20 minutes a solution of 0.10 g of N,N'-ethylene-bis-acrylamide in 5.0 g of a mixture of t-butanol and water (90% t-butanol by weight) was injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles with sizes from 10 μ m to 300 μ m was obtained with a solid content of approximately 13% by weight. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 9

[0052] Test composition 4 was prepared in accordance with the following procedure. A mixture of 90.0 g of a mixture of t-butanol and water (90% t-butanol by weight), 15.0 g of acrylamide, 0.50 g of poly(vinyl pyrrolidone), was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. After approximately

20 minutes a solution of 0.40 g of N,N'-ethylene-bis-acrylamide in 5.0 g of a mixture of t-butanol and water (90% t-butanol by weight) was injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles was obtained with a solid content of approximately 13% by weight. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 10

[0053] Test composition 5 was prepared in accordance with the following procedure. A mixture of 90.0 g of a mixture of t-butanol and water (90% t-butanol by weight), 15.0 g of acrylamide, 0.50 g of poly(vinyl pyrrolidone), was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. After approximately 20 minutes a solution of 0.20 g of N,N'-ethylene-bis-acrylamide in 5.0 g of a mixture of t-butanol and water (90% t-butanol by weight) was injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles was obtained with a solid content of approximately 13% by weight. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 11

[0054] Test composition 6 was prepared in accordance with the following procedure. A mixture of 90.0 g of a mixture of t-butanol and water (90% t-butanol by weight), 15.0 g of acrylamide, 0.50 g of poly(vinyl pyrrolidone), was prepared in a 250 mL three-neck flask equipped with a condenser and a mechanical stirrer. The mixture was purged with nitrogen gas for 30 minutes and then heated to 65°C. A 5.0 g solution of azobisisobutyronitrile in ethanol (1% azobisisobutyronitrile by weight) was then injected into the mixture. After approximately 20 minutes a solution of 0.050 g of N,N'-ethylene-bis-acrylamide in 5.0 g of a mixture of t-

butanol and water (90% t-butanol by weight) was injected into the mixture. The mixture was then kept at 65°C under nitrogen and stirred for 22 hours at 300 rpm. The mixture was then allowed to cool to room temperature and a dispersion of particles was obtained with a solid content of approximately 13% by weight. The dispersions were then centrifuged at 4800 rpm for 5 minutes. The supernatant was then decanted and sediments were collected. The sediments were then derived under vacuum at approximately 50°C overnight to generate loose, well dispersed dried powders.

Example 12

[0055] Test composition 7 was prepared in accordance with the following procedure. Equal amounts of the powders from Examples 8, 9, 10, and 11 were mixed to generate a powdered fluid loss control additive.

Example 13

[0056] Fluid loss tests were performed on test compositions 1-7 in accordance with the following procedure. Varying dosages of each of the test compositions as well as conventional fluid loss control additive N-Drill HT Plus® (commercially available from Halliburton Energy Services, Inc., Houston, Texas) were added to separate drilling mud compositions comprising 0.09 g of viscosifier N-VIS® (commercially available from Halliburton Energy Services, Inc., Houston, Texas), 14.0 g of bridging agent BARACARB® 5, (commercially available from Halliburton Energy Services, Inc., Houston, Texas) 3.5 g of bridging agent CARACARB® 5 (commercially available from Halliburton Energy Services, Inc., Houston, Texas), and 137 g of a 26% by weight solution of NaCl in water. The amount of NaCl in the drilling fluids that the fluid loss control additives from Examples 6 and 7 were added to was reduced to account for the brine present in those fluid loss control additives. The drilling fluids were each placed into a 175 mL "OFITE" HPHT Filter Press for fluid loss evaluation. In a typical experiment, pressures were adjusted to 600 and 100 psi (700 and 200 psi when the temperature is above 300°F) on the top and bottom of a sample chamber, which was filled with mud and preheated to test temperature. Drained liquid, from a filter paper with a porosity of 2.7 µm sealed on the bottom of the cylinder, was collected. The performance of each fluid loss control additive is provided below in Table 1. The acceptable performance by an additive was set to be 10 ML volume of filtrate with 30 min test.

Table 1

Fluid Loss Control Additive	Temperature (°F)	Crosslinking Agent (wt %)	Dosage (wt %)	Fluid Loss (mL)
N-DRILL HT PLUS®	250	N/A	2.0	9.5
Test Composition 1	250	0	2.0	100
Test Composition 2	250	0.006	2.0	4.3
N-DRILL HT PLUS®	300	N/A	2.0	100
Test Composition 2	300	0.006	2.0	100
Test Composition 2	300	0.006	1.0	32
Test Composition 2	300	0.006	0.5	37
Test Composition 3	300	0.7	2.0	6.3
Test Composition 3	300	0.7	1.0	16.3
Test Composition 3	300	0.7	0.5	50.0
Test Composition 3	350	0.7	2.0	40.0
Test Composition 4	350	2.7	2.0	10.2
Test Composition 5	350	1.3	2.0	7.5
Test Composition 6	350	0.3	2.0	43.5
Test Composition 7	350	1.3	2.0	6.5

[0057] In some embodiments, the present invention provides a method comprising: providing an aqueous based treatment fluid comprising a fluid loss control additive that comprises at least one polymeric micro gel; placing the aqueous based treatment fluid in a subterranean formation via a well bore penetrating the subterranean formation; allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation; allowing the filter cake to be degraded; and producing hydrocarbons from the formation.

[0058] As can be seen by Table 1, test composition 1 which represented a liner high molecular weight polymer did not show effectiveness as nearly all fluid drained out. Test composition 2 (in brine solution) was found to perform better than N-Drill HT Plus® (fluid loss of 4.3 mL compared to a fluid loss of 9.5 mL) at the same dosage at 250°F. However, when tested at 300°F, both test composition 2 and N-DRILL HT Plus® nearly failed completely during

the test at a dosage of 2 weight percent. Test composition 2 was able to reduce fluid loss to 32 mL and 37 mL at a dosage of 1 weight percent and 0.5 weight percent, respectively.

[0059] At 300°F, test composition 3 (crosslinking agent content of approximately 0.7 weight percent) controlled fluid loss around 6.3 mL, however this additive failed to meet the criteria of acceptable performance of 10 mL volume of filtrate within 30 minutes at 350°F. Test compositions 5 and 7 (crosslinking agent content of approximately 1.3 weight percent) were able to control the fluid loss around 7.5 and 6.5 mL at 350°F, respectively. However, increasing the crosslinking agent content to 2.7 weight percent (as demonstrated by composition 4) resulted in fluid loss of 10.2 mL.

[0060] As can be seen by Table 1, and further demonstrated by Figure 3, dosage has an effect on the fluid loss control. As shown in Figure 3, the fluid loss by test composition 3 decreased from 50 mL to 6.3 mL as the dosage of this additive increased from 0.5 wt percent to 2.0 weight percent. Thus, it is highly likely that the fluid loss would be further reduced at dosages higher than 2.0 weight percent, even though an optimal dosage might exist.

[0061] As can further be seen by Table 1, the filtrate volume decreased from 43.5 mL to 7.5 mL, when the crosslinking agent content of the corresponding fluid loss additive increased from 0.3 weight percent to 1.3 weight percent (See test compositions 6, 3, and 5). With a further increase of the crosslinking agent content to 2.7 weight percent (see test composition 4), the filtrate volume increased slightly to 10.2 mL. Surprisingly, synergist effect was observed when a mixture was used as the additive (See test composition 7). The mud containing this additive led to a fluid loss of only 6.5 mL, lower than those by any other non-mixed test compositions and well below the acceptable value of 10 mL. Without wishing to be limited to theory, it is believed that the synergy was probably due to the physical distribution of the polymeric micro gel particles in the mud, which enhanced the fluid loss reduction.

[0062] Therefore, the present invention is well adapted to attain the ends and advantages mentioned as well as those that are inherent therein. The particular embodiments disclosed above are illustrative only, as the present invention may be modified and practiced in different but equivalent manners apparent to those skilled in the art having the benefit of the teachings herein. Furthermore, no limitations are intended to the details of construction or design herein shown, other than as described in the claims below. It is therefore evident that the particular illustrative embodiments disclosed above may be altered or modified and all such

variations are considered within the scope and spirit of the present invention. All numbers and ranges disclosed above may vary by some amount. Whenever a numerical range with a lower limit and an upper limit is disclosed, any number and any included range falling within the range are specifically disclosed. In particular, every range of values (of the form, "from about a to about b," or, equivalently, "from approximately a to b," or, equivalently, "from approximately a-b") disclosed herein is to be understood to set forth every number and range encompassed within the broader range of values. Moreover, the indefinite articles "a" or "an", as used in the claims, are defined herein to mean one or more than one of the element that it introduces. Also, the terms in the claims have their plain, ordinary meaning unless otherwise explicitly and clearly defined by the patentee.

What is claimed is:

1. A drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel.

2. The drilling fluid of claim 1 wherein the fluid loss control additive is included in the drilling fluid in an amount up to about 5% by weight of active component of the polymeric micro gel.

3. The drilling fluid of claim 1 wherein the polymeric micro gel has a diameter in the range of about 10 nm to about 1 mm.

4. The drilling fluid of claim 1 wherein the polymeric micro gel comprises a reaction product formed by a polymerization reaction of a polymer or copolymer and a crosslinking agent.

5. The drilling fluid of claim 4 wherein the polymer or copolymer comprises at least one unit based on at least one compound selected from the group consisting of: acrylamides, vinyl alcohols, vinylpyrrolidones, vinylpyridines, acrylates, polyacrylamides, polyvinyl alcohols, polyvinylpyrrolidones, polyvinylpyridines, polyacrylates, polybutylene succinate, polybutylene succinate-co-adipate, polyhydroxybutyrate-valerate, polyhydroxybutyrate-covalerate, polycaprolactones, polyester amides, polyethylene terephthalates, sulfonated polyethylene terephthalate, polyethylene oxides, polyethylenes, polypropylenes, aliphatic aromatic copolyester, polyacrylic acids, polysaccharides (such as dextran or cellulose), chitins, chitosans, proteins, aliphatic polyesters, polylactic acids, poly(glycolides), poly(ϵ -caprolactones), poly(hydroxy ester ethers), poly(hydroxybutyrates), poly(anhydrides), polycarbonates, poly(orthoesters), poly(amino acids), poly(ethylene oxides), poly(propylene oxides), poly(phosphazenes), polyester amides, polyamides, polystyrenes, a copolymer thereof, a homopolymer thereof, a tetrapolymer thereof, and any derivative thereof.

6. The drilling fluid of claim 4 wherein the crosslinking agent comprises at least one crosslinking agent selected from the group consisting of: diethylene glycol dimethacrylate, azobisisobutyronitrile, and 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride.

7. The drilling fluid of claim 1 further comprising a bridging agent.

8. A method comprising:

providing an aqueous based treatment fluid comprising a fluid loss control additive that comprises at least one polymeric micro gel;

placing the aqueous based treatment fluid in a subterranean formation via a well bore penetrating the subterranean formation;

allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation;

allowing the filter cake to be degraded; and

producing hydrocarbons from the formation.

9. The method of claim 8 wherein the fluid loss control additive is included in the aqueous based treatment fluid in an amount up to about 5% by weight of active component of the polymeric micro gel.

10. The method of claim 8 wherein the polymeric micro gel has a diameter in the range of about 10 nm to about 1 mm.

11. The method of claim 8 wherein the polymeric micro gel comprises a reaction product formed by a polymerization reaction of a polymer or copolymer and a crosslinking agent.

12. The method of claim 11 wherein the polymer or copolymer comprises at least one unit based on at least one compound selected from the group consisting of: acrylamides, vinyl alcohols, vinylpyrrolidones, vinylpyridines, acrylates, polyacrylamides, polyvinyl alcohols, polyvinylpyrrolidones, polyvinylpyridines, polyacrylates, polybutylene succinate, polybutylene succinate-co-adipate, polyhydroxybutyrate-valerate, polyhydroxybutyrate-covalerate, polycaprolactones, polyester amides, polyethylene terephthalates, sulfonated polyethylene terephthalate, polyethylene oxides, polyethylenes, polypropylenes, aliphatic aromatic copolyester, polyacrylic acids, polysaccharides (such as dextran or cellulose), chitins, chitosans, proteins, aliphatic polyesters, polylactic acids, poly(glycolides), poly(ϵ -caprolactones), poly(hydroxy ester ethers), poly(hydroxybutyrates), poly(anhydrides), polycarbonates, poly(orthoesters), poly(amino acids), poly(ethylene oxides), poly(propylene oxides), poly(phosphazenes), polyester amides, polyamides, polystyrenes, a copolymer thereof, a homopolymer thereof, a tetrapolymer thereof, derivatives and any combinations thereof.

13. The method of claim 11 wherein the crosslinking agent comprises at least one crosslinking agent selected from the group consisting of: 2,2'-azobis-(2-methylbutyronitrile), 2,2'-azobis(isobutyramidine hydrochloride), 2,2'-azobis[2-(2-imidazolin-2-

yl)propane]dihydrochloride, 1,1'-azobis(cyclohexanecarbonitrile), 2,2'-azobis(2-methylpropionamidine) dihydrochloride, 4,4'-azobis(4-cyanovaleic acid), ammonium persulfate, hydroxymethanesulfinic acid monosodium salt dihydrate, potassium persulfate, sodium persulfate, benzoyl peroxide, 1,1-bis(*tert*-amylperoxy)cyclohexane, 1,1-bis(*tert*-butylperoxy)cyclohexane, 2,2-bis(*tert*-butylperoxy)butane, 2,4-pentanedione peroxide, 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane, 2,5-di(*tert*-butylperoxy)-2,5-dimethyl-3-hexyne; 2-butanone peroxide, cumene hydroperoxide, di-*tert*-amyl peroxide, dicumyl peroxide, lauroyl peroxide, *tert*-butyl hydroperoxide, *tert*-butyl peracetate, *tert*-butyl peroxide, *tert*-butyl peroxybenzoate, *tert*-butylperoxy-2-ethylhexyl carbonate, diethylene glycol dimethacrylate, azobisisobutyronitrile, derivatives and combinations thereof.

14. The method of claim 8 wherein the aqueous based treatment fluid further comprises a bridging agent.

15. A method comprising:
 providing a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel;
 circulating the drilling fluid in a subterranean formation;
 allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation;
 allowing the filter cake to be degraded; and
 producing hydrocarbons from the formation.

16. The method of claim 15 wherein the fluid loss control additive is included in the drilling fluid in an amount up to about 5% by weight of active component of the polymeric micro gel.

17. The method of claim 15 wherein the polymeric micro gel has a diameter in the range of about 10 nm to about 1 mm.

18. The method of claim 15 wherein the polymeric micro gel comprises a reaction product formed by a polymerization reaction of a polymer or copolymer and a crosslinking agent.

19. The method of claim 18 wherein the polymer or copolymer comprises at least one unit based on at least one compound selected from the group consisting of: acrylamides, vinyl alcohols, vinylpyrrolidones, vinylpyridines, acrylates, polyacrylamides,

polyvinyl alcohols, polyvinylpyrrolidones, polyvinylpyridines, polyacrylates, polybutylene succinate, polybutylene succinate-co-adipate, polyhydroxybutyrate-valerate, polyhydroxybutyrate-covalerate, polycaprolactones, polyester amides, polyethylene terephthalates, sulfonated polyethylene terephthalate, polyethylene oxides, polyethylenes, polypropylenes, aliphatic aromatic copolyester, polyacrylic acids, polysaccharides (such as dextran or cellulose), chitins, chitosans, proteins, aliphatic polyesters, polylactic acids, poly(glycolides), poly(ϵ -caprolactones), poly(hydroxy ester ethers), poly(hydroxybutyrates), poly(anhydrides), polycarbonates, poly(orthoesters), poly(amino acids), poly(ethylene oxides), poly(propylene oxides), poly(phosphazenes), polyester amides, polyamides, polystyrenes, a copolymer thereof, a homopolymer thereof, a tetrapolymer thereof, derivatives and combinations thereof.

20. The method of claim 18 wherein the crosslinking agent comprises at least one crosslinking agent selected from the group consisting of: 2,2'-azobis(2-methylbutyronitrile), 2,2'-azobis(isobutyramidine hydrochloride), 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride, 1,1'-azobis(cyclohexanecarbonitrile), 2,2'-azobis(2-methylpropionamidine) dihydrochloride, 4,4'-azobis(4-cyanovaleric acid), ammonium persulfate, hydroxymethanesulfinic acid monosodium salt dihydrate, potassium persulfate, sodium persulfate, benzoyl peroxide, 1,1-bis(*tert*-amylperoxy)cyclohexane, 1,1-bis(*tert*-butylperoxy)cyclohexane, 2,2-bis(*tert*-butylperoxy)butane, 2,4-pentanedione peroxide, 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane, 2,5-di(*tert*-butylperoxy)-2,5-dimethyl-3-hexyne; 2-butanone peroxide, cumene hydroperoxide, di-*tert*-amyl peroxide, dicumyl peroxide, lauroyl peroxide, *tert*-butyl hydroperoxide, *tert*-butyl peracetate, *tert*-butyl peroxide, *tert*-butyl peroxybenzoate, *tert*-butylperoxy-2-ethylhexyl carbonate, diethylene glycol dimethacrylate, azobisisobutyronitrile, derivatives and combinations thereof.

21. The method of claim 15 wherein the drilling fluid further comprises a bridging agent.

22. A method comprising:

providing an inside screen pill comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel, and

placing the inside screen pill in a subterranean formation so that the inside screen pill is located between a gravel pack screen and an area of the subterranean formation.

23. The method of claim 22 wherein the fluid loss control additive is included in the inside screen pill in an amount of about 1% to about 10% by weight of active component of the polymeric micro gel.

24. The method of claim 22 wherein the polymeric micro gel has a diameter in the range of about 10 nm to about 1 mm.

25. The method of claim 22 wherein the polymeric micro gel comprises a reaction product formed by a polymerization reaction of a polymer or copolymer and a crosslinking agent.

26. The method of claim 25 wherein the crosslinking agent comprises at least one crosslinking agent selected from the group consisting of: 2,2'-azobis-(2-methylbutyronitrile), 2,2'-azobis(isobutyramidine hydrochloride), 2,2'-azobis[2-(2-imidazolin-2-yl)propane]dihydrochloride, 1,1'-azobis(cyclohexanecarbonitrile), 2,2'-azobis(2-methylpropionamidine) dihydrochloride, 4,4'-azobis(4-cyanovaleric acid), ammonium persulfate, hydroxymethanesulfinic acid monosodium salt dihydrate, potassium persulfate, sodium persulfate, benzoyl peroxide, 1,1-bis(*tert*-amylperoxy)cyclohexane, 1,1-bis(*tert*-butylperoxy)cyclohexane, 2,2-bis(*tert*-butylperoxy)butane, 2,4-pentanedione peroxide, 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane, 2,5-di(*tert*-butylperoxy)-2,5-dimethyl-3-hexyne; 2-butanone peroxide, cumene hydroperoxide, di-*tert*-amyl peroxide, dicumyl peroxide, lauroyl peroxide, *tert*-butyl hydroperoxide, *tert*-butyl peracetate, *tert*-butyl peroxide, *tert*-butyl peroxybenzoate, *tert*-butylperoxy-2-ethylhexyl carbonate, diethylene glycol dimethacrylate, azobisisobutyronitrile, derivatives and combinations thereof.

27. The method of claim 25 wherein the polymer or copolymer comprises at least one unit based on at least one compound selected from the group consisting of: acrylamides, vinyl alcohols, vinylpyrrolidones, vinylpyridines, acrylates, polyacrylamides, polyvinyl alcohols, polyvinylpyrrolidones, polyvinylpyridines, polyacrylates, polybutylene succinate, polybutylene succinate-co-adipate, polyhydroxybutyrate-valerate, polyhydroxybutyrate-covalerate, polycaprolactones, polyester amides, polyethylene terephthalates, sulfonated polyethylene terephthalate, polyethylene oxides, polyethylenes, polypropylenes, aliphatic aromatic copolyester, polyacrylic acids, polysaccharides (such as dextran or cellulose), chitins, chitosans, proteins, aliphatic polyesters, polylactic acids, poly(glycolides), poly(ϵ -caprolactones), poly(hydroxy ester ethers), poly(hydroxybutyrates),

poly(anhydrides), polycarbonates, poly(orthoesters), poly(amino acids), poly(ethylene oxides), poly(propylene oxides), poly(phosphazenes), polyester amides, polyamides, polystyrenes, a copolymer thereof, a homopolymer thereof, a tetrapolymer thereof, and any derivative thereof.

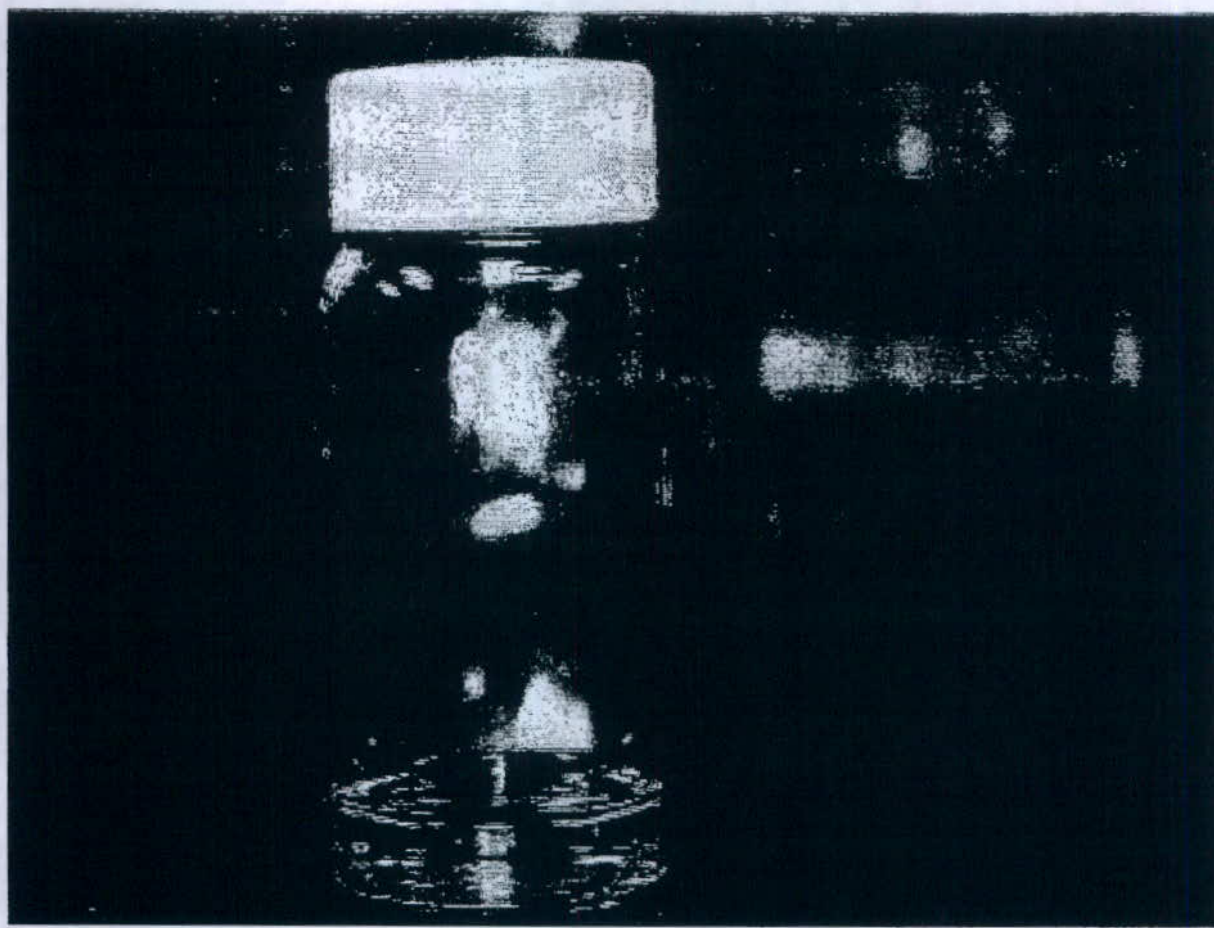


Figure 1



Figure 2

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2010/000657

A. CLASSIFICATION OF SUBJECT MATTER
INV. C09K8/512
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 182 417 A (GIVENS STEVEN P [US] ET AL) 8 January 1980 (1980-01-08) claims 1,2,7; table 2 the whole document	1-7
X	US 2008/135247 A1 (HUTCHINS RICHARD D [US]) 12 June 2008 (2008-06-12) * abstract paragraphs [0008], [0015], [0020], [022], [0028] - [0032] the whole document	1,3-8, 10-15, 17-21
X	GB 2 278 359 A (SOFITECH NV [BE]) 30 November 1994 (1994-11-30) page 6, line 5 - page 7, line 19 the whole document	1,2,4-7
	----- -/--	

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents:

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- *G* document member of the same patent family

Date of the actual completion of the international search

11 August 2010

Date of mailing of the international search report

17/08/2010

Name and mailing address of the ISA/

European Patent Office, P.O. 5818 Patentlaan 2
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Authorized officer

Straub, Thomas

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2010/000657

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2008/142220 A1 (HOEFER ANN M W [US] ET AL) 19 June 2008 (2008-06-19) paragraphs [0016] - [0021] the whole document -----	1-6, 22-27
X	US 2008/096774 A1 (TABARY RENE [FR] ET AL) 24 April 2008 (2008-04-24) paragraphs [0034], [0047], [0060], [0071] - [0077] the whole document -----	22-27
X	US 2007/032386 A1 (ABAD CARLOS [US] ET AL) 8 February 2007 (2007-02-08) the whole document -----	1-5, 8-12, 15-19, 22-25, 27

INTERNATIONAL SEARCH REPORT

International application No.
PCT/GB2010/000657

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☒ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- ☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- ☒ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. claims: 1-7

A drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel.

2. claims: 8-14

A method comprising:
 providing an aqueous based treatment fluid comprising a fluid loss control additive that comprises at least one polymeric micro gel;
 placing the aqueous based treatment fluid in a subterranean formation via a well bore penetrating the subterranean formation;
 allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation;
 allowing the filter cake to be degraded; and
 producing hydrocarbons from the formation.

3. claims: 15-21

A method comprising:
 providing a drilling fluid comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel;
 circulating the drilling fluid in a subterranean formation;
 allowing the fluid loss control additive to become incorporated into a filter cake located on a surface within the subterranean formation;
 allowing the filter cake to be degraded; and
 producing hydrocarbons from the formation.

4. claims: 22-27

A method comprising:
 providing an inside screen pill comprising an aqueous base fluid and a fluid loss control additive that comprises at least one polymeric micro gel, and
 placing the inside screen pill in a subterranean formation so that the inside screen pill is located between a gravel pack screen and an area of the subterranean formation.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2010/000657

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 4182417	A	08-01-1980	NONE	
US 2008135247	A1	12-06-2008	CA 2611047 A1	12-06-2008
GB 2278359	A	30-11-1994	EP 0705850 A1	10-04-1996
			FR 2704219 A1	28-10-1994
US 2008142220	A1	19-06-2008	NONE	
US 2008096774	A1	24-04-2008	AR 051561 A1	24-01-2007
			CA 2577940 A1	09-03-2006
			EP 1799960 A2	27-06-2007
			FR 2874617 A1	03-03-2006
			WO 2006024795 A2	09-03-2006
US 2007032386	A1	08-02-2007	CA 2662210 A1	27-03-2008
			EA 200970299 A1	30-10-2009
			WO 2008035254 A2	27-03-2008
			US 2010163228 A1	01-07-2010

PATENT COOPERATION TREATY

From the RECEIVING OFFICE

PCT

NOTIFICATION OF THE INTERNATIONAL APPLICATION NUMBER AND OF THE INTERNATIONAL FILING DATE

(PCT Rule 20.2(c))

To:
A A Thornton & Co
235 High Holborn
LONDON

WC1V 7LE

Date of mailing
(day/month/year) 13 APR 2010

Applicant's or agent's file reference PAC/N/27915		IMPORTANT NOTIFICATION	
International application No. PCT/GB2010/000657	International filing date (day/month/year) 01/04/2010	Priority date (day/month/year) 03/04/2009	
Applicant Halliburton Energy Services, Inc. et al			
Title of the invention Methods Of Using Fluid Loss Additives Comprising Micro Gells			

1. The applicant is hereby notified that the international application has been accorded the international application number and the international filing date indicated above.

2. The applicant is further notified that the record copy of the international application:

☒ was transmitted to the International Bureau on 13 APR 2010

☐ has not yet been transmitted to the International Bureau for the reason indicated below and a copy of this notification has been sent to the International Bureau*:

☐ because the necessary national security clearance has not yet been obtained.

☐ because (reason to be specified):

* The International Bureau monitors the transmittal of the record copy by the receiving Office and will notify the applicant (with Form PCT/IB/301) of its receipt. Should the record copy not have been received by the expiration of 14 months from the priority date, the International Bureau will notify the applicant (Rule 22.1(c)).

Name and mailing address of the receiving Office Intellectual Property Office Cardiff Road, Newport South Wales NP10 8QQ	Authorized officer BRIGITTE SPARKES
Facsimile No.	Telephone No. 01633 814033

Form PCT/RO/105 (July 2008)

Intellectual Property Office is an operating name of the Patent Office

133

PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference (if desired) (12 characters maximum) PAC/N/27915

Box No. I TITLE OF INVENTION	
METHODS OF USING FLUID LOSS ADDITIVES COMPRISING MICRO GELLS	
Box No. II APPLICANT ☐ This person is also inventor	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)	Telephone No.
HALLIBURTON ENERGY SERVICES, INC. P O Box 1431 Duncan Oklahoma 73536 US	Facsimile No.
	Applicant's registration No. with the Office
State (that is, country) of nationality: US	State (that is, country) of residence: US
This person is applicant for the purposes of: ☐ all designated States ☒ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box	
Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)	
☒ Further applicants and/or (further) inventors are indicated on a continuation sheet.	
Box No. IV AGENT OR COMMON REPRESENTATIVE: OR ADDRESS FOR CORRESPONDENCE	
The person identified below is hereby has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: ☒ agent ☐ common representative	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)	Telephone No.
CURTIS, Philip, Anthony. A A Thornton & Co 235 High Holborn London WC1V 7LE GB	+44 20 7405 4044
	Facsimile No.
	+44 20 7405 3580
	Agent's registration No. with the Office
☐ Address for correspondence: Mark this check-box where no agent or common representative is has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.	

134

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S) <i>If none of the following sub-boxes is used, this sheet should not be included in the request.</i>	
Name and address: <i>(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</i> EZELL, Ryan, G. 2367 Village Leaf Drive Spring Texas 77386 US	This person is: ☐ applicant only ☒ applicant and inventor ☐ inventor only <i>(If this check-box is marked, do not fill in below.)</i> Applicant's registration No. with the Office
State <i>(that is, country)</i> of nationality: US	State <i>(that is, country)</i> of residence: US
This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box	
Name and address: <i>(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</i> WU, Jun, Jim, 9014 Landsdown Drive Houston Texas 77096 US	This person is: ☐ applicant only ☒ applicant and inventor ☐ inventor only <i>(If this check-box is marked, do not fill in below.)</i> Applicant's registration No. with the Office
State <i>(that is, country)</i> of nationality: CA	State <i>(that is, country)</i> of residence: US
This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box	
Name and address: <i>(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</i> CURTIS, Philip, Anthony, A A Thornton & Co 235 High Holborn London WC1V 7LE GB	This person is: ☒ applicant only ☐ applicant and inventor ☐ inventor only <i>(If this check-box is marked, do not fill in below.)</i> Applicant's registration No. with the Office
State <i>(that is, country)</i> of nationality: GB	State <i>(that is, country)</i> of residence: GB
This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☐ the United States of America only ☒ the States indicated in the Supplemental Box	
Name and address: <i>(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</i> 	This person is: ☐ applicant only ☐ applicant and inventor ☐ inventor only <i>(If this check-box is marked, do not fill in below.)</i> Applicant's registration No. with the Office
State <i>(that is, country)</i> of nationality: 	State <i>(that is, country)</i> of residence:
This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box	
☐ further applicants and/or (further) inventors are indicated on another continuation sheet.	

Supplemental Box

If the Supplemental Box is not used, this sheet should not be included in the request.

1 If, in any of the Boxes, except Boxes Nos. VIII(ii) to (iv) for which a special continuation box is provided, the space is insufficient to furnish all the information; in such case, write "Continuation of Box No. ..." indicate the number of the Box) and furnish the information in the same manner as required according to the captions of the Box in which the space was insufficient, in particular:

CONTINUATION OF BOX III

P A Curtis is applicant for Malawi (MW) only.

(i) if more than one person is to be indicated as applicant and/or inventor and no "continuation sheet" is available; in such case, write "Continuation of Box No. III" and indicate for each additional person the same type of information as required in Box No. III. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below:

CONTINUATION OF BOX NO IV

Further agents of A A Thornton & Co., 235 High Holborn, London, WC1V 7LE, United Kingdom, who have been appointed to act on behalf of the applicants before the competent International Authorities are:

(ii) if, in Box No. II or in any of the sub-boxes of Box No. III, the indication "the States indicated in the Supplemental Box" is checked: in such case, write "Continuation of Box No. II" or "Continuation of Box No. III" or "Continuation of Boxes No. II and No. III" (as the case may be), indicate the name of the applicant(s) involved and, next to each such name, the State(s) (and/or, where applicable, ARIPO, Eurasian, European or OAPI patent) for the purposes of which the named person is applicant;

LERWILL, John,
GILL, Ian Stephen,
HEDGES, Martin Nicholas,
TURNER, Craig Robert,
BENNETT, Adrian Robert James,
SOUTH, Nicholas Geoffrey
JENNINGS, Michael John
COTTRILL, Emily Elizabeth Helen
PATEL, Nikesh Prakash
AMERY, Marcus James

(iii) if, in Box No. II or in any of the sub-boxes of Box No. III, the inventor or the inventor/applicant is not inventor for the purposes of all designated States or for the purposes of the United States of America: in such case, write "Continuation of Box No. II" or "Continuation of Box No. III" or "Continuation of Boxes No. II and No. III" (as the case may be), indicate the name of the inventor(s) and, next to each such name, the State(s) (and/or, where applicable, ARIPO, Eurasian, European or OAPI patent) for the purposes of which the named person is inventor;

(iv) if, in addition to the agent(s) indicated in Box No. IV, there are further agents: in such case, write "Continuation of Box No. IV" and indicate for each further agent the same type of information as required in Box No. IV;

(v) if, in Box No. VI, there are more than three earlier applications whose priority is claimed: in such case, write "Continuation of Box No. VI" and indicate for each additional earlier application the same type of information as required in Box No. VI.

2 If the applicant intends to make an indication of the wish that the international application be treated, in certain designated States, as an application for a patent of addition, certificate of addition, inventor's certificate of addition or utility certificate of addition: in such a case, write the name or two-letter code of each designated State concerned and the indication "patent of addition," "certificate of addition," "inventor's certificate of addition" or "utility certificate of addition," the number of the parent application or parent patent or other parent grant and the date of grant of the parent patent or other parent grant or the date of filing of the parent application (Rules 4.11(a)(iii) and 49bis.1(a) or (b)).

3 If the applicant intends to make an indication of the wish that the international application be treated, in the United States of America, as a continuation or continuation-in-part of an earlier application: in such a case, write "United States of America" or "US" and the indication "continuation" or "continuation-in-part" and the number and the filing date of the parent application (Rules 4.11(a)(iv) and 49bis.1(d)).

Box No. V DESIGNATIONS				
The filing of this request constitutes under Rule 4.9(a), the designation of all Contracting States bound by the PCT on the international filing date, for the grant of every kind of protection available and, where applicable, for the grant of both regional and national patents. However, ☐ DE: Germany is not designated for any kind of national protection ☐ JP: Japan is not designated for any kind of national protection ☐ KR: Republic of Korea is not designated for any kind of national protection ☐ RU: Russian Federation is not designated for any kind of national protection <i>(The check-boxes above may only be used to exclude (irrevocably) the designations concerned if, at the time of filing or subsequently under Rule 26bis.1, the international application contains in Box No. VI a priority claim to an earlier national application filed in the particular State concerned, in order to avoid the ceasing of the effect, under the national law, of this earlier national application.)</i>				
Box No. VI PRIORITY CLAIM				
The priority of the following earlier application(s) is hereby claimed:				
Filing date of earlier application <i>(day:month:year)</i>	Number of earlier application	Where earlier application is:		
		national application: country or Member of WTO	regional application: regional Office	international application: receiving Office
item (1) 03/04/09 (3 APRIL 2009)	12/418,323	US		
item (2)				
item (3)				
☐ Further priority claims are indicated in the Supplemental Box.				
Transmit certified copy: the receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) <i>(only if the earlier application was filed with the Office which for the purposes of this international application is the receiving Office)</i> identified above as: ☐ all items ☐ item (1) ☐ item (2) ☐ item (3) ☐ other, see Supplemental Box				
Restore the right of priority: the receiving Office is requested to restore the right of priority for the earlier application(s) identified above or in the Supplemental Box as item(s) (). <i>(See also the Notes to Box No. VI: further information must be provided to support a request to restore the right of priority.)</i>				
Incorporation by reference: where an element of the international application referred to in Article 11(1)(iii)(d) or (e) or a part of the description, claims or drawings referred to in Rule 20.5(a) is not otherwise contained in this international application but is completely contained in an earlier application whose priority is claimed on the date on which one or more elements referred to in Article 11(1)(iii) were first received by the receiving Office, that element or part is, subject to confirmation under Rule 20.6, incorporated by reference in this international application for the purposes of Rule 20.6.				
Box No. VII INTERNATIONAL SEARCHING AUTHORITY				
Choice of International Searching Authority (ISA) <i>if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used:</i> ISA: EP				
Request to use results of earlier search: reference to that search <i>if an earlier search has been carried out by or requested from the International Searching Authority</i> Date <i>(day:month:year)</i> Number Country <i>(or regional Office)</i>				
Box No. VIII DECLARATIONS				
The following declarations are contained in Boxes Nos. VIII (i) to (v) <i>(mark the applicable check-boxes below and indicate in the right column the number of each type of declaration):</i>				Number of declarations
☐	Box No. VIII (i)	Declaration as to the identity of the inventor	:	
☐	Box No. VIII (ii)	Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent	:	
☐	Box No. VIII (iii)	Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application	:	
☐	Box No. VIII (iv)	Declaration of inventorship (only for the purposes of the designation of the United States of America)	:	
☐	Box No. VIII (v)	Declaration as to non-prejudicial disclosures or exceptions to lack of novelty	:	

Box No. IX CHECK LIST: LANGUAGE OF FILING	
This international application contains: (a) on paper, the following number of sheets: request (including declaration and supplemental sheets) : 5 description (excluding sequence listing and/or tables related thereto) : 22 claims : 6 abstract : 1 drawings : 2 Sub-total number of sheets : 36 sequence listing : tables related thereto : (for both, actual number of sheets if filed on paper, whether or not also filed in electronic form; see (c) below) Total number of sheets : 36 (b) ☐ only in electronic form (Section 801(a)(i)) (i) ☐ sequence listing (ii) ☐ tables related thereto (c) ☐ also in electronic form (Section 801(a)(ii)) (i) ☐ sequence listing (ii) ☐ tables related thereto Type and number of carriers (diskette, CD-ROM, CD-R or other) on which are contained the ☐ sequence listing: ☐ tables related thereto: (additional copies to be indicated under items 9(ii) and/or 10(ii), in right column)	This international application is accompanied by the following item(s) (mark the applicable check-boxes below and indicate in right column the number of each item): 1. ☒ fee calculation sheet : 1 2. ☐ original separate power of attorney : 3. ☐ original general power of attorney : 4. ☐ copy of general power of attorney; reference number, if any: : 5. ☐ statement explaining lack of signature : 6. ☐ priority document(s) identified in Box No. VI as item(s): : 7. ☐ translation of international application into (language): : 8. ☐ separate indications concerning deposited microorganism or other biological material : 9. ☐ sequence listing in electronic form (indicate type and number of carriers) (i) ☐ copy submitted for the purposes of international search under Rule 13ter only (and not as part of the international application) : (ii) ☐ (only where check-box (b)(i) or (c)(ii) is marked in left column) additional copies including, where applicable, the copy for the purposes of international search under Rule 13ter : (iii) ☐ together with relevant statement as to the identity of the copy or copies with the sequence listing mentioned in left column : 10. ☐ tables in electronic form related to sequence listing (indicate type and number of carriers) (i) ☐ copy submitted for the purposes of international search under Section 802(b-quater) only (and not as part of the international application) : (ii) ☐ (only where check-box (b)(ii) or (c)(ii) is marked in left column) additional copies including, where applicable, the copy for the purposes of international search under Section 802(b-quater) : (iii) ☐ together with relevant statement as to the identity of the copy or copies with the tables mentioned in left column : 11. ☒ other (specify): Letter : 1
Figure of the drawings which should accompany the abstract: Fig 1	Language of filing of the international application: English
Box No. X SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request). CURTIS, Philip, Anthony. 1 APRIL 2010	

For receiving Office use only	
1. Date of actual receipt of the purported international application:	2. Drawings: ☐ received: ☐ not received:
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application.	
4. Date of timely receipt of the required corrections under PCT Article 17(2):	
5. International Searching Authority (if two or more are competent): ISA :	6. ☐ Transmittal of search copy delayed until search fee is paid

For International Bureau use only
Date of receipt of the record copy by the International Bureau:



No. 11-149158-00000-0000

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Act. 411 PRESENTACION Folios: 141

LISTA DE CHEQUEO
ADMISIÓN A TRÁMITE – NUEVAS CREACIONES

~~Art. 411 PRESENTACION~~ Folios: 138
PATENTE DE INVENCION ☐

MODELO DE UTILIDAD ☐

Art 33 Decisión 486/00

☐	Indicación que se solicita una patente.
☐	Datos de identificación del solicitante o de la persona que presenta la solicitud
☐	Descripción de la invención
☐	Dibujos de ser estos pertinentes
☐	Comprobante de pago de las tasas establecidas (De ser el caso formato de descuento)
Completa ☐	Incompleta ☐

PATENTE DE INVENCION PCT ☒ MODELO DE UTILIDAD PCT ☐ Art.33 Decisión 486/00, Circular Única

☒	Indicación que se solicita una PCT
☒	Copia de la solicitud en español, tal como fue presentada inicialmente (capítulo descriptivo, reivindicatorio, resumen)
☒	Dibujos de ser estos pertinentes
☒	Comprobante de pago de las tasas establecidas (de ser el caso formato de descuento)
Completa ☒	Incompleta ☐

DISEÑO INDUSTRIAL ☐ (Art. 119 Decisión 486/00)

☐	Indicación que se solicita Diseño industrial
☐	Datos de identificación del solicitante o de la persona que presenta la solicitud
☐	Representación gráfica y fotográfica del Diseño industrial o muestra del material que incorpora el diseño
☐	Comprobante de pago de las tasas establecidas
Completa ☐	Incompleta ☐

ESQUEMA DE TRAZADO ☐ (Art. 92 Decisión 486/00)

☐	Indicación que se solicita un esquema de trazado
☐	Datos de identificación del solicitante o de la persona que presenta la solicitud
☐	Representación gráfica de un esquema de trazado
☐	Comprobante de pago de las tasas establecidas
Completa ☐	Incompleta ☐