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(54) Benævnelse: **MINIEMULSIONPOLYMERISERING TIL FREMSTILLING AF MODSTANDSREDUKTIONSMIDLER**

(56) Fremdragne publikationer:

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G. QI, C.W. JONES, F.J. SCHORK: "RAFT inverse miniemulsion polymerization of acrylamide", MACROMOLECULAR RAPID COMMUNICATIONS, vol. 28, 2 May 2007 (2007-05-02), pages 1010-1016, XP002698684,

DESCRIPTION

FIELD OF THE INVENTION

[0001] A method of producing a polymer latex drag reducer from a miniemulsion.

BACKGROUND OF THE INVENTION

[0002] A drag reducer is a composition capable of substantially reducing friction loss associated with the turbulent flow of a fluid through a conduit. Where fluids are transported over long distances, such as in oil and other hydrocarbon liquid pipelines, these friction losses result in inefficiencies that increase equipment and operations costs. Ultra-high molecular weight polymers are known to function well as drag reducers, particularly in hydrocarbon liquids. In general, drag reduction depends in part upon the molecular weight of the polymer additive and its ability to dissolve in the hydrocarbon under turbulent flow. Effective drag-reducing polymers typically have molecular weights in excess of five million.

[0003] In the past, it has been proposed that drag reducers comprising polymeric latex emulsions can be used to reduce friction loss associated with turbulent fluid flow commonly been proposed for application to the flow of hydrocarbon streams (e.g., crude oil, gasoline, diesel fuel, etc.) through pipelines. In order to be most effective, the drag reducer must be dissolved in the hydrocarbon stream.

[0004] Currently, a number of different commercial approaches are being taken to address the problem of preparing, dissolving, transporting and using such drag reducing polymers. A common commercial method is to prepare the polymer in dilute solutions in an inert solvent such as kerosene or other solvating material. This method utilizes a solution of high molecular weight polymer suitable for use as a drag reducing agent when produced by polymerization of alpha olefins in a hydrocarbon solvent. The entire mixture, containing polyolefin, solvent, and catalyst particles is used without separation to form dilute solutions of the polymer in crude oil or finished hydrocarbons.

[0005] Another current commercial approach to drag reduction is to polymerize alpha olefins in bulk form, mill the bulk polymer into small (50μ to 500μ) particles, and then suspend the particles in an aqueous or other (non-solvent) liquid medium. This method allows for much higher levels of active drag reducing polymer in the final product (up to about 25%).

[0006] Yet another method is to polymerize monomers with slight water solubility using emulsion polymerization. This method has the advantage that suspensions containing high levels of active drag reduction polymer (up to 45%) can be prepared. Unfortunately, the types of monomers that can be used in emulsion polymerization are not always the best choices for making drag reducing polymers for use in hydrocarbon fluids (crude oil, gasoline, and diesel fuel).

[0007] The use of miniemulsion polymerization techniques for the production of drag recuing polymer prepared with very water insoluble monomers solves the problem of using the best, water insoluble monomers for the production of drag reducers for use in hydrocarbon liquids.

[0008] Document US 2006/148928 relates to a polymerization process to produce a drag reducer that can have in some embodiments a molecular weight of at least 5 million. The process is illustrated in example 1 in which a solution comprising water, a surfactant and a buffer is prepared and mixed with a solution comprising a monomer (2-ethylhexyl methacrylate). A redox initiator is used and added to the mixed solution.
Document WO 2005/090851 relates to the use of nanoemulsions as polymeric drag reducers. The nanoemulsions are prepared by combining the component parts with agitation and/or mixing sufficient to form water-soluble polymer/water droplets of acceptably small size.

SUMMARY OF THE INVENTION

[0009] A method of producing a polymer latex drag reducer. The method begins by forming an aqueous solution comprising a surfactant, a buffer and water. The method then forms an organic solution comprising a monomer and a co-stabilizer. The aqueous solution and the organic solution are mixed to form an emulsion. The emulsion is then subjected to a high shear device to produce a miniemulsion, wherein the monomers are broken into small droplets followed by polymerizing the miniemulsion with the addition of an initiator, wherein a nucleation occurs in the small monomer droplets.

DETAILED DESCRIPTION OF THE INVENTION

[0010] The present method provides a method to produce a polymer latex drag reducer. The method begins with forming an aqueous solution with a surfactant, a buffer and water in addition to forming an organic solution from a monomer and a co-stabilizer. The aqueous solution and the organic solution are mixed to form an emulsion. The emulsion is then subjected to a high shear device such as a sonifier to produce a miniemulsion, wherein the monomers are broken into small droplets. The miniemulsion is then polymerized with the addition of an initiator comprising an oxidizer and a reducer, wherein the nucleation occurs in the small droplets. The initiator can be either water soluble or oil soluble and is only added after subjecting the emulsion ot a high shear device. The initiator can also be produced by mixing an oxidizer and a reducer or even, a secondary buffer, an oxidizer and a reducer. The polymerization process produces a polymer latex drag reducer.

[0011] There are many benefits that can be attributed to using a miniemulsion to produce a drag reducing product. One benefit is the enhanced ability to mix water insoluble monomers together. Since nucleation occurs in small monomer droplets it is possible for the monomers to be more thoroughly mixed together. A second benefit is increased drag reducing properties. The drag reducing properties can improve 5, 10, 15, 20, 25, 30 35, 40, or even 50% by using the miniemulsion process. The drag reducing properties of the polymer do not affect the viscosity of the crude oil, more specifically heavy crude oil that it is placed into. Instead the drag reducing polymers are able to break up the turbulent flow in the pipelines that the heavy crude oil flows in, thereby improving the flow of the oil in the pipeline. Another benefit that miniemulsion polymerization provide is that water insoluble monomer do not need to transfer from monomer droplets to micelles to start polymerization as in conventional emulsion polymerization. The step of monomer diffusion is eliminated.

[0012] Suitable examples of heavy crude oils include, but are not limited to, Merey heavy crude, Petrozuata heavy crude, Corocoro heavy crude, Albian heavy crude, Bow River heavy crude, Maya heavy crude, and San Joaquin Valley heavy crude. Additionally, the crude oil can be a blend of heavy crude oil with lighter hydrocarbons or diluents. Suitable examples of blended crude oils include, but are not limited to, Western Canadian Select and Marlim Blend.

[0013] The aqueous solution contains a surfactant, a buffer and water which are stirred together. The resultant homogeneous aqueous solution can have the following properties.

Reactant	Broad Range	Narrow Range
Surfactant (wt% of aqueous solution)	0-20%	0.002-5%
Buffer (wt% of aqueous solution)	0-20%	0.001-5%
Water (wt% of aqueous solution)	50-100%	80-99.99%

[0014] The surfactant used can include at least one high HLB anionic or nonionic surfactant. The term "HLB number" refers to the hydrophile-lipophile balance of a surfactant in an emulsion. The HLB number is determined by the methods described by W.C. Griffin in J. Soc. Cosmet. Chem., 1, 311 (1949) and J. Soc. Cosmet. Chem., 5, 249 (1954). As used herein, the term "high HLB" shall denote an HLB number of 7 or more. The HLB number of surfactants for use with forming the reaction mixture can be at least about 8, at least about 10, or at least 12.

[0015] Exemplary high HLB anionic surfactants include, but are not limited to, high HLB alkyl sulfates, alkyl ether sulfates, dialkyl sulfosuccinates, alkyl phosphates, alkyl aryl sulfonates, and sarcosinates. Suitable examples of commercially available high HLB anionic surfactants include, but are not limited to, sodium lauryl sulfate (available as RHODAPON LSB from Rhodia Incorporated, Cranbury, NJ), dioctyl sodium sulfosuccinate (available as AEROSOL OT from Cytec Industries, Inc., West Paterson, NJ), 2-ethylhexyl polyphosphate sodium salt (available from Jarchem Industries Inc., Newark, NJ), sodium dodecylbenzene sulfonate (available as NORFOX 40 from Norman, Fox & Co., Vernon, CA), and sodium lauroylsarcosinic (available as HAMPOSYL L-30 from Hampshire Chemical Corp., Lexington, MA).

[0016] Exemplary high HLB nonionic surfactants include, but are not limited to, high HLB sorbitan esters, PEG fatty acid esters, ethoxylated glycerine esters, ethoxylated fatty amines, ethoxylated sorbitan esters, block ethylene oxide/propylene oxide surfactants, alcohol/fatty acid esters, ethoxylated alcohols, ethoxylated fatty acids, alkoxylated castor oils, glycerine esters, linear alcohol ethoxylates, and alkyl phenol ethoxylates. Suitable examples of commercially available high HLB nonionic surfactants include, but are not limited to, nonylphenoxy and octylphenoxy poly(ethyleneoxy) ethanols (available as the IGEPAL CA and CO series, respectively from Rhodia, Cranbury, NJ), C8 to C18 ethoxylated primary alcohols (such as RHODASURF LA-9 from Rhodia Inc., Cranbury, NJ), C11 to C15 secondary-alcohol ethoxylates (available as the TERGITOL 15-S series, including 15-S-7, 15-S-9, 15-S-12, from Dow Chemical Company, Midland, MI), polyoxyethylene sorbitan fatty acid esters (available as the TWEEN series of surfactants from Uniqema, Wilmington, DE), polyethylene oxide (25) oleyl ether (available as SIPONIC Y-500-70 from Americal Alcolac Chemical Co., Baltimore, MD), alkylaryl polyether alcohols (available as the TRITON X series, including X-100, X-165, X-305, and X-405, from Dow Chemical Company, Midland, MI).

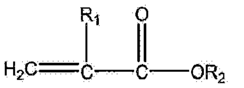
[0017] The buffer can comprise any known buffer that is compatible with the initiation system such as, for example, carbonate, phosphate, and/or borate buffers. Specific buffers that can be used include potassium dihydrogen phosphate and dipotassium hydrogen phosphate.

[0018] The organic solution contains a monomer and a co-stabilizer which are stirred together. The resultant homogeneous organic solution have the following properties costabilizer are totally dissolved in the monomer

Reactant	Broad Range	Narrow Range
Monomer (wt% of organic solution)	80-99.99%	90-99%
Co-stabilizer (wt% of organic solution)	0.01-20%	1-10%

[0019] In one embodiment of the present invention, the drag reducing polymer can comprise a plurality of repeating units of the residues of one or more of the monomers selected from the group consisting of:

(A)



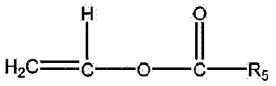
$$H_2C=C(R_1)-C(=O)-OR_2$$

wherein R_1 is H or a C1-C10 alkyl radical, and R_2 is H, a C1-C30 alkyl radical, a C5-C30 substituted or unsubstituted cycloalkyl radical, a C6-C20 substituted or unsubstituted aryl radical, an aryl-substituted C1-C10 alkyl radical, a -(CH2CH2O)$_x$-R_A or -(CH2CH(CH3)O)$_x$-R_A radical wherein x is in the range of from 1 to 50 and R_A is H, a C1-C30 alkyl radical, or a C6-C30 alkylaryl radical;

(B) R_3-arene-R_4

wherein arene is a phenyl, naphthyl, anthracenyl, or phenanthrenyl, R_3 is $CH{=}CH_2$ or $CH_3{-}C{=}CH_2$, and R_4 is H, a C1-C30 alkyl radical, a C5-C30 substituted or unsubstituted cycloalkyl radical, Cl, SO_3, OR_B, or $COOR_C$, wherein R_B is H, a C1-C30 alkyl radical, a C5-C30 substituted or unsubstituted cycloalkyl radical, a C6-C20 substituted or unsubstituted aryl radical, or an aryl-substituted C1-C10 alkyl radical, and wherein R_C is H, a C1-C30 alkyl radical, a C5-C30 substituted or unsubstituted cycloalkyl radical, a C6-C20 substituted or unsubstituted aryl radical, or an aryl-substituted C1-C10 alkyl radical;

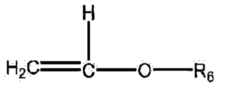
(C)



$$H_2C{=}C(H){-}O{-}C({=}O){-}R_5$$

wherein R_5 is H, a C1-C30 alkyl radical, or a C6-C20 substituted or unsubstituted aryl radical;

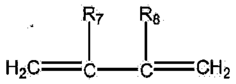
(D)



$$H_2C{=}C(H){-}O{-}R_6$$

wherein R_6 is H, a C1-C30 alkyl radical, or a C6-C20 substituted or unsubstituted aryl radical;

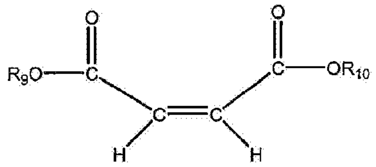
(E)



$$H_2C{=}C(R_7){-}C(R_8){=}CH_2$$

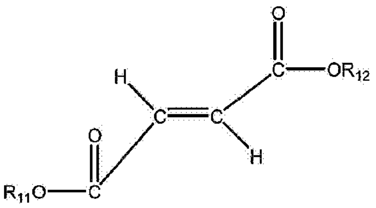
wherein R_7 is H or a C1-C18 alkyl radical, and R_8 is H, a C1-C18 alkyl radical, or Cl;

(F)



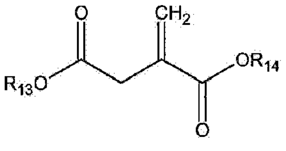
wherein R_9 and R_{10} are independently H, a C1-C30 alkyl radical, a C6-C20 substituted or unsubstituted aryl radical, a C5-C30 substituted or unsubstituted cycloalkyl radical, or heterocyclic radicals;

(G)



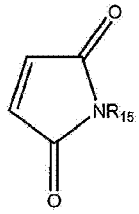
wherein R_{11} and R_{12} are independently H, a C1-C30 alkyl radical, a C6-C20 substituted or unsubstituted aryl radical, a C5-C30 substituted or unsubstituted cycloalkyl radical, or heterocyclic radicals;

(H)



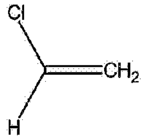
wherein R_{13} and R_{14} are independently H, a C1-C30 alkyl radical, a C6-C20 substituted or unsubstituted aryl radical, a C5-C30 substituted or unsubstituted cycloalkyl radical, or heterocyclic radicals;

(I)

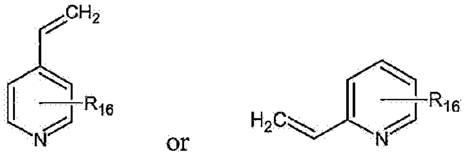


wherein R_{15} is H, a C1-C30 alkyl radical, a C6-C20 substituted or unsubstituted aryl radical, a C5-C30 substituted or unsubstituted cycloalkyl radical, or heterocyclic radicals;

(J)

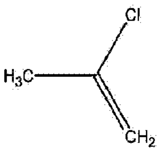


(K)



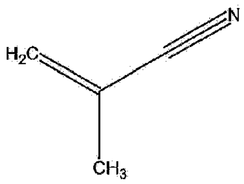
wherein R_{16} is H, a C1-C30 alkyl radical, or a C6-C20 aryl radical;

(L)

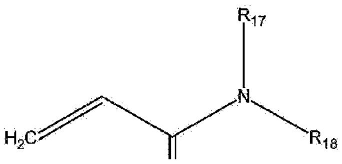


(M)

(N)

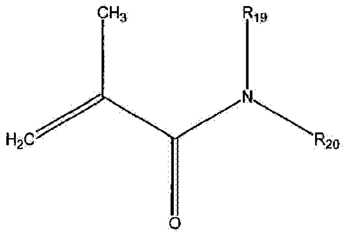


(O)



wherein R_{17} and R_{18} are independently H, a C1-C30 alkyl radical, a C6-C20 substituted or unsubstituted aryl radical, a C5-C30 substituted or unsubstituted cycloalkyl radical, or heterocyclic radicals;

(P)



wherein R_{19} and R_{20} are independently H, a C1-C30 alkyl radical, a C6-C20 substituted or unsubstituted aryl radical, a C5-C30 substituted or unsubstituted cycloalkyl radical, or heterocyclic radicals.

[0020] In one embodiment of the present invention, the drag reducing polymer can comprise repeating units of the residues of C4-C20 alkyl, C6-C20 substituted or unsubstituted aryl, or aryl-substituted C1-C10 alkyl ester derivatives of methacrylic acid or acrylic acid. In another embodiment, the drag reducing polymer can be a copolymer comprising repeating units of the residues of 2-ethylhexyl methacrylate and the residues of at least one other monomer. In yet another embodiment, the drag reducing polymer can be a copolymer comprising repeating units of the residues of 2-ethylhexyl methacrylate monomers and butyl acrylate monomers. In still another embodiment, the drag reducing polymer can be a homopolymer comprising repeating units of the residues of 2-ethylhexyl methacrylate.

[0021] In one embodiment of the present invention, the drag reducing polymer can comprise the residues of at least one monomer having a heteroatom. As stated above, the term "heteroatom" includes any atom that is not a carbon or hydrogen atom. Specific examples of heteroatoms include, but are not limited to, oxygen, nitrogen, sulfur, phosphorous, and/or chlorine atoms. In one embodiment, the drag reducing polymer can comprise at least about 10 percent, at least about 25 percent, or at least 50 percent of the residues of monomers having at least one heteroatom. Additionally, the heteroatom can have a partial charge. As used herein, the term "partial charge" is defined as an electric charge, either positive or negative, having a value of less than 1.

[0022] The co-stabilizer can be any highly water insoluble small molecules that are presently known in the art. Examples of co-stabilizers that can be used include fatty alcohols (C_{12}-C_{20}), hexadecane, isohexadecane and hydrophobic oligomers such as styrene. Some of the most widely used co-stabilizers include cetyl alcohol and hexadecane. The co-stabilizer is required in to effectively stabilize the high shearing that is required to form the miniemulsion. Due to the high solubility of co-stabilizer in water insoluble monomer, so-stabilizer prevents monomer particles from coagulation and stabilizes the monomer particles after shearing.

[0023] The organic solution and aqueous solution are then mixed together to form an emulsion. The emulsion is then subjected to a high-shear device. High shear devices can be any commonly known device in the art such as sonifier, microfluidizers, static mixer or homogenizers. The high shear devices should be able to produce submicron dispersions of monomer droplets whose droplet sizes can range from 20 to 1000 nm or even 50 to 500 nm. By reducing the droplet sizes the primary loci of nucleation would occur in the droplets rather than the micelles. This increases the possibility that polymerization of the hydrophobic monomers would take place in the monomer droplets, which in turn allows the miniemulsion to prepare oil-soluble polymers as drag reducer products.

[0024] The miniemulsion is then polymerized with the addition of an initiator. The initiator can comprise of only an oxidizer and a reducer or it can contain a secondary buffer, an oxidizer and a reducer. The initiator can also be aqueous.

Reactant	Broad Range	Narrow Range
Secondary Buffer (wt% of polymer)	0-20%	0-5%
Oxidizer (wt% of polymer)	0-1%	0-0.1%
Reducer	0-1%	0-0.02%

[0025] The secondary buffer can be any commonly used buffer. Ideal buffers are those used described above that were used in production of the homogeneous aqueous solution.

[0026] The oxidizer solution can be any commonly used oxidizer solution. In one embodiment the oxidizing solution are persulfates, peroxides or azonitrile initiators. Possible persulfate oxidizers that can be used include ammonium persulfate, sodium persulfate and potassium persulfate. Possible peroxides that can be used include hydrogen peroxide and organic peroxides.

[0027] The reducer is selected to reduce the oxidizer. In one embodiment the reducer is a salt. The reducer can be sulfites such as bisulfite or hydrosulfite, acids such as ascorbic acid or erythrobic acid, iron based reducers or even sodium formaldehyde sulfoxylate. One example of a reducer that is frequently used is ferrous ammonium sulfate, hexahydrate, $Fe(NH_4)_2(SO_4)_2{\cdot}6H_2O$).

[0028] One optional component is a secondary surfactant. The secondary surfactant can be any commonly used surfactant. Ideal surfactants are those used described above that were used in production of the homogeneous aqueous solution.

Examples

[0029] Calculation of drag reduction properties. Pipeline field testing was performed with various diameter pipelines, and various crude oils. For each of the tests the percent drag reduction (%DR) was determined by measuring the pressure drop in the segment of pipe being tested prior to addition of drag reducing agent (ΔPbase) and measuring the pressure drop in the segment of pipe being tested after addition of drag reducing agent (ΔP¬¬treated). The percent drag reduction was then determined according to the following formula:

%DR = ((ΔPbase ¬– ΔPtreated) / ΔPbase) x 100%

Example 1

Batch Polymerization - 300-mL Reactor

Polymer reactants

[0030]

Component	Recipe (grams)	±	Actual (g)
Deionized Water	93.88	0.20	93.90
Polystep B-5	7.52	0.05	7.53
Tergitol 15-S-7	8.00	0.05	8.00
Phosphate buffer (87.0 g potassium dihydrogen phosphate 68.0 g potassium hydrogen phosphate in 1000 mL deionized water)	2.60	0.02	2.60
Ammonium persulfate solution	4.00	0.02	4.02
2-Ethylhexyl methacrylate	80.00	0.20	80.00

Oxidizer solution

[0031]

Component	Weight (g)	±	Actual (g)
Ammonium persulfate, $(NH_4)_2S_2O_8$	0.133	0.005	0.1331
Deionized Water	40.00	0.05	40.02

[0032] The emulsion was done in a reactor for four hours under nitrogen at a temperature set around 2.0°C. The agitator was set at 400 rpm. A catalyst solution was then added to the emulsion when the temperature was 5 °C.

Catalyst (Iron) solution:

[0033]

Component	Weight (g)	±	Actual (g)
Ferrous ammonium sulfate, hexahydrate, $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$,	0.357	0.004	0.3573
0.010 M sulfuric acid solution (3.71 grams concentrated sulfuric acid in 3785 mL deionized water)	50.00	0.05	50.00

[0034] The catalyst solution was injected over 21 hours at 188 µL/hr with a total volume injected of 3.18 mL.

[0035] The final product weight of the polymer was 200.92 grams with the following characteristics.

sample #	Wet Weight (g)	Dry Weight (g)	% Solid	Ave. % Solid	Ave. % Polymer
GLT-8220-203R-a	1.2308	0.5311	43.15	42.81	37.53
GLT-8220-203R-b	1.2424	0.5277	42.47		

sample #	Theo. % Solid	Theo. % Polymer	% conversion
GLT-8220-203R	45.27	39.99	93.85

[0036] The drag reduction property of the polymer was tested at 28.8% at 2 ppm.

Example 2

Batch Polymerization - 300-mL Reactor

Aqueous solution

[0037]

Component	Recipe (grams)	±	Actual
Deionized Water	93.90	0.50	93.91
Polystep B-5	4.32	0.02	4.33
Tergitol 15-S-7	8.00	0.02	8.00
Phosphate buffer (87.0 g potassium dihydrogen phosphate 68.0 g potassium hydrogen phosphate in 1000 mL DI water)	2.60	0.02	2.61

Organic solution

[0038]

Component	Recipe (grams)	±	Actual
Ethylhexyl methacrylate	80.00	0.50	80.05
Cetyl alcohol	3.20	0.02	3.21

[0039] The aqueous solution and the organic solution where then mixed together and subjected to a high shear device. The jacket temperature set to around 2.0°C and the agitator at 400 rpm. The emulsion was sheared under nitrogen for 1 hour.

Oxidizer solution

[0040]

Component	Weight (g)	±	Actual
Ammonium persulfate, $(NH_4)_2S_2O_8$	0.133	0.001	0.133
Deionized Water	40.00	0.05	40.01

[0041] The oxidizer solution was injected into the miniemulsion at 0.10 mL/minute over 40 minutes while still under nitrogen.

Catalyst (Iron) solution:

[0042]

Component	Weight (g)	±	Actual
Ferrous ammonium sulfate, hexahydrate, $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$	0.1428	0.001	0.1428
0.010 M sulfuric acid solution (3.71 grams concentrated sulfuric acid in 3785 mL deionized water)	200.0	0.05	200.00

[0043] The catalyst (iron) solution was injected into the miniemulsion when the miniemulsion was 5°C. The syringe pump was programmed to deliver at 188 µL/hr for 21 hours a total of 2.91 mL of the catalyst.

[0044] The final product weight of the polymer was 187.52 grams with the following characteristics.

sample #	Wet Weight (g)	Dry Weight (g)	% Solid	Ave. % Solid	Ave. % Polymer
GLT8220-61	2.2937	1.0780	47.00	47.06	40.26
	2.2736	1.0710	47.11		

sample #	Theo. % Solid	Theo. % Polymer	% conversion
GLT8220-61	49.22	42.42	94.91

[0045] The drag reduction property of the polymer was tested at 36.4% at 2 ppm.

Example 3

Batch Polymerization - 300-mL Reactor

Polymer reactants

[0046]

Component	Recipe (grams)	±	Actual
Deionized Water	93.88	0.20	93.88
Polystep B-5	7.52	0.05	7.52
Tergitol 15-S-7	8.00	0.05	8.01
Phosphate buffer (87.0 g potassium dihydrogen			

Component	Recipe (grams)	±	Actual
phosphate 68.0 g potassium hydrogen phosphate in 1000 mL DI water)	2.60	0.02	2.60
Ammonium persulfate solution	4.00	0.02	4.00
Isodecyl methacrylate	80.00	0.20	80.05

Oxidizer solution

[0047]

Component	Weight (g)	±	Actual
Ammonium persulfate, $(NH_4)_2S_2O_8$	0.133	0.005	0.133
Deionized Water	40.00	0.05	40.00

[0048] The reactor was purged for four hours under nitrogen at a temperature set around 5.0°C before starting polymerization. The agitator was set at 400 rpm. A catalyst solution was then added to the emulsion when the temperature was 5 °C.

Catalyst (Iron) solution:

[0049]

Component	Weight (g)	±	Actual
Ferrous ammonium sulfate, hexahydrate, $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$	0.1428	0.001	0.1428
0.010 M sulfuric acid solution(3.71 grams concentrated sulfuric acid in 3785 mL DI water)	400.0	0.05	400.01

[0050] The catalyst solution was injected over 24 hours at 330 µL/hr with a total volume injected of 4.92mL.

[0051] The final product weight of the polymer was 197.97 grams with the following characteristics.

sample #	Wet Weight (g)	Dry Weight (g)	% Solid	Ave. % Solid	Ave. % Polymer
GLT8220-104	1.5202	0.6725	44.24	44.27	38.97
	1.5206	0.6737	44.30		

sample #	Theo. % Solid	Theo. % Polymer	% conversion
GLT8220-104	45.53	40.23	96.87

[0052] The drag reduction property of the polymer was tested at 0% at 2 ppm.

Example 4

Batch Polymerization - 300-mL Reactor

Aqueous solution

[0053]

Component	(Recipe) grams	±	Actual
Deionized Water	97.90	0.50	97.90
Polystep B-5	4.30	0.02	4.30
Phosphate buffer (87.0 g potassium dihydrogen phosphate 68.0 g potassium hydrogen phosphate in 1000 mL DI water)	2.60	0.02	2.61

Organic solution

[0054]

Component	Recipe (grams)	±	Actual
Isodecyl methacrylate	80.00	0.50	80.00
Hexadecane	3.20	0.02	3.20

[0055] The aqueous solution and the organic solution where then mixed together for 10 minutes and subjected to a high shear device. The high shear device used was a Branson sonifier at the output power setting of eight and a duty cycle of 50%. After sonification the solution was cooled down to around 5.0°C placed under nitrogen for 1 hour

Oxidizer solution

[0056]

Component	Weight (g)	±	Actual
Ammonium persulfate, $(NH_4)_2S_2O_8$	0.133	0.001	0.1330
Deionized Water	40.00	0.05	40.00

[0057] The oxidizer solution was injected into the miniemulsion at 0.10 mL/minute over 40 minutes while still under nitrogen.

Catalyst (Iron) solution:

[0058]

Component	Weight (g)	±	Actual
Ferrous ammonium sulfate, hexahydrate, $Fe(NH_4)_2(SO_4)_2\cdot 6H_2O$	0.1428	0.001	0.1449
0.010 M sulfuric acid solution (3.71 grams concentrated sulfuric acid in 3785 mL deionized water)	400.0	0.05	400.00

[0059] The catalyst (iron) solution was injected into the miniemulsion when the miniemulsion was 5°C. The syringe pump was programmed to deliver at 330 µL/hr for 24 hours a total of 7.71 mL of the catalyst.

[0060] The final product weight of the polymer was 196.60 grams with the following characteristics.

sample #	Wet Weight (g)	Dry Weight (g)	% Solid	Ave. % Solid	Ave. % Polymer
GLT-8391-048L-a	1.7202	0.7002	40.70	40.68	38.24
GLT-8391-048L-b	1.6791	0.6828	40.66		

sample #	Theo. % Solid	Theo. % Polymer	% conversion
GLT-8391-048L	42.93	40.49	94.44

[0061] The drag reduction property of the polymer was tested at 19.25% at 2 ppm.

REFERENCES CITED IN THE DESCRIPTION

Patent documents cited in the description

- US2006148928A [0008]
- WO2005090851A [0008]

Non-patent literature cited in the description

- **W.C. GRIFFIN**J. Soc. Cosmet. Chem., 1949, vol. 1, 311- [0014]
- J. Soc. Cosmet. Chem., 1954, vol. 5, 249- [0014]

MINIEMULSIONPOLYMERISERING TIL FREMSTILLING AF MODSTANDSREDUKTIONSMIDLER

PATENTKRAV

1. Fremgangsmåde til dannelse af et latexpolymermodstandsmiddel, der omfatter:

dannelse af en vandig opløsning, der omfatter et overfladeaktivt middel, en buffer og vand;

dannelse af en organisk opløsning, der omfatter en monomer og et costabiliseringsmiddel;

blanding af den vandige opløsning og den organiske opløsning for at danne en emulsion;

udsættelse af emulsionen for en anordning med høj forskydning for at frembringe en miniemulsion, hvor monomerne er brudt op i små dråber; og

polymerisering af miniemulsionen ved tilsætning af en initiator, hvor initiatoren omfatter et oxideringsmiddel og et reduktionsmiddel, hvor der sker kernedannelse i de små monomerdråber;

hvor polymeriseringen frembringer et latexpolymermodstandsmiddel; og

hvor initiatoren først tilsættes efter udsættelse af emulsionen for en anordning med høj forskydning.

2. Fremgangsmåde ifølge krav 1, hvor costabiliseringsmidlet er udvalgt fra gruppen bestående af cetylalkohol, hexadecan og kombinationer deraf.

3. Fremgangsmåde ifølge krav 1, hvor anordningen med høj forskydning anvendes til at frembringe dråber i intervallet fra 50-500 nm.

4. Fremgangsmåde ifølge krav 1, hvor anordningen med høj forskydning omfatter en sonifikeringsanordning, en mikrofluidiseringsanordning, en statisk blander eller en homogeniseringsanordning.

5. Fremgangsmåde ifølge krav 1, hvor initiatoren er vandig.

6. Fremgangsmåde ifølge krav 1, hvor initiatoren endvidere omfatter en sekundær buffer.

7. Fremgangsmåde ifølge krav 1, hvor den vandige opløsning og den organiske opløsning er homogene.