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(54) Title: COATED PROPPANT AND METHOD OF PROPPANT FLOWBACK CONTROL

(57) Abstract: The invention relates to oil and gas production industry and can be used for stimulation of production well by preventing of fracture closure by pumping of proppants during hydraulic fracturing treatment of pay zones.



WO 2009/088315 A1

## **COATED PROPPANT AND METHOD OF PROPPANT FLOWBACK CONTROL**

This invention relates to the gas and oil production industry and can be used for stimulation of production well thorough sustaining of hydraulic fractures by application of propping granules – proppants – during hydraulic fracturing treatment for the oil pay zones.

A serious problem in oil production is the flowback of proppant from the fracture back towards the wellbore: this happens after hydraulic fracturing of formation, during the first fracture cleanup, and sometimes after well completion. The literature data show that up to 20 % of the pumped proppant is usually washed away from a fracture during fracture cleanup and production; this causes several adverse consequences. In wells with a low production rate, the removed proppant may deposit on the casing; this situation requires regular cleanup and makes costly the repair operations. Another problem that can be caused by high proppant flowback rate is a failure or fast depreciation of electrical submersible pumps (ESP). Proppant flowback reduces the fracture conductivity due to fracture thickness loss; this reduces the well production rate.

There exist several known methods for proppant flowback control.

The most available approach is using a curable resin coated proppant (US3492147, US3929191, US5218038, US5639806) that is pumped into the fracture at the end of fracturing treatment. The use of this proppant has several drawbacks caused by-side chemical reactions of the resin coating with the fracturing fluid. On one hand, these reactions result in degradation and deteriorating of the resin coating integrity, reduces the strength of bond between the proppant particulate, which, in turn, reduces the proppant packing strength. On the other hand, chemical reactions between the resin coating and components

of hydraulic fracturing fluid produce uncontrollable changes in fluid rheology and undermine the efficiency of fracturing treatment. These factors and cyclic loading (closing/opening well operations) and long-run period of well shut-off may deteriorate the proppant packing strength.

Chemical compounds of the resin coating may interact with the gel breaker (e.g., ammonium persulfate) and this declines the actual concentration of the gel breaker. This is detrimental for the fracture cleanup aimed to removing of remnants of the polymer gel and this reduces the fracture permeability.

In some case, e.g., in the event of rapid closure of the fracture, the resin proppant has not time to solidify and get the nominal strength. This increases the yield of crushed proppant and reduces the fracture permeability.

The use of dual resin coated particles (patents US4585064, US4717594) partially alleviates this problem. The inner resin coating is substantially cured, and the outer coating consists of fusible resin material. The inner layer of the cured resin provides the required strength of the proppant under high external loads and increases the crush resistance of side substrate. The second or outer coating of a heat curable resin has a potential for curing with the neighboring proppant particles and produces a cohesive permeable mass.

The method is known (patents US4888240, US5422183, US5597784), when strengthening of the resin coating and the interface between the inner/outer resin coating and particulate/resin interface is achieved by reinforcing agents and composites: mineral fillers (talc, silica flour, mica) or polymers selected from the group consisting of polyisobutylene, ethylene-vinyl acetate copolymers, and ethylene-propylene copolymers. The reinforcing agent is deposited after coating the particle with the inner resin coating but before the inner coating is cured. A second resin coating is then formed over the inner resin resulting in a high strength particle having the reinforcing agent interspersed in the inner resin

coating/outer resin coating boundary.

As it was mentioned above, the serious drawback of resin-coated proppants is poor compatibility with the fracturing fluids and with polymer breakers. The patents US5837656 and US5955144 describe improved resin coated particles having better compatibility of resin with chemical components of the fracturing fluid. They offer the same concept of dual resin coating, but change the order of the curable and substantially cured resin. This achieved by creating of particle with a thin cured resin coating that is compatible with fracturing fluids encapsulating an inner coating of a curable resin. Such particles do not affect fracturing properties and still consolidate into a strong permeable mass when subjected to subterranean condition. The first inner coating attached to the proppant particle is a fusible curable composition, preferably novolac resin. The second, protective, outer coating on said particulate substrate is preferably made of a heat-curable resole resin. The lab tests demonstrated a good compatibility of the packing to consolidation under downhole conditions.

The further development of the concept of resin-coated proppant is described in the patent US7153575 (the difference from patents US5837656, US 5955144 is that the particulate is coated with two and more curable coatings of resin).

The development of the known method is described in patents US7135231, patent application US2007/0036977. Multilayer proppant (made from number n additional resin coats onto the proppant) comprises of series of incrementally applied resin microlayer coating such that each of the microlayer partial coatings are interleaved with each other. The patent also describes the method of the proppant production which is disclosed in the special sputtering technology of resin deposition. "Sputtering" technology refers to a physical deposition method that does not involve solution based chemistry, wherein the chemicals to be

deposited are generally used as a feedstock and fed into air or gas-powder nozzle which is aimed at the surface of substrate.

Patents US6114410 and US6328105 describe an improved proppant and a method of increasing fracture conductivity in subterranean formations. The proppant contains a mixture of bondable and removable particles. The bondable particles can be coated with a curable resin. The bondable particles within a subterranean formation adhere to adjacent bondable particles to form a permanent, self-supporting matrix; and the removable particles from the self-supporting matrix provide the ambient fracture conditions. This increases fracture conductivity and the overall productivity of the hydraulic operation.

Известен способ, при котором для ограничения выноса пропантa используют частицы покрытые дополнительным слоем состоящим из эластомерных материалов (US2006/0035790), а также их комбинации полимеров с волокнами (US7244492).

The patents US7244492, US2006/0035790 disclosure resin coating proppant with additional elastomeric and fibrous materials coatings.

The patent US5330005 describes the method for proppant flowback control through mixing of a regular proppant with fiber material. Fibers intermingle with proppant pack and reduce flowback. Besides the strengthening of the proppant pack, added fibers redistribute the loads, making bridges on the most part of proppant pack area. A fiber-hold structure is more flexible than that composed of resin coated proppant: it allows small shifts in the proppant-fiber packing without loss in strength.

A mechanism of using deformable, thermoplastic, and elastomeric materials for proppant flowback control is based on embedment of proppant particulate into deformable material. Patent US6059034 describes deformable beaded particulate. Deformable particulate facilitates effective redistribution of

stresses inside the packing, and improves the packing strength due to a higher contact area between the particles. A serious drawback of this soft material in a significant reduction in the free pore space in particulate packing because particles penetrate the pores and reduce the fracture permeability and, ultimately, the well production.

A partial solution for this problem (US6330916) is production of core-and-coating particulate. The core material of deformable particulate material comprising at least one of ground or crushed nut shells, ground or crushed seed shells, ground or crushed fruit pits, processed wood, or a mixture thereof. The coating material is polymeric compounds.

A mixture of proppant with adhesive polymer materials can be used for proppant flowback control (US5582249). Adhesive material comes in contact with proppant and makes a thin and tacking coating. This material facilitates adhesion between particulate and sand or/and crashed fines; this stops completely or partially the proppant flowback from the fracture. The typical feature of adhesive coating is that particles remain tacky for a long time even at elevated downhole temperatures without cross-linking or solidifying.

The patent US7032667 discloses about fracture propping with use of tacky agents and resin-coated proppant.

The patent US6742590 discloses the method of proppant flowback control by mixing of tacky materials with deformable particles (every component is already effective tool for flowback control).

Another kind of material suitable for proppant flowback control is thermoplastic materials (US5501274, EP0735235). Thermoplastic compound is mixed with proppant, then it melts at a higher subterranean temperature and sticks to proppant; this creates aggregates of adhered proppant.

The patent US5697440 describes the method of application of

thermoplastic material with resin-coated proppant.

The patent US6830105 discloses about the method for proppant flowback control, wherein the thermoplastic elastomer is mixed with proppant as a liquid (or a solution with appropriate solvent). Then the dissolved elastomer is cured independently or with curing agent producing a thermoplastic coating.

Patent application US2006/0169448 proposes a new fracturing fluid composition which is self-degrading cement consisting of an acid component and a base component whose interaction results in formation of the cement material, and of a degrading component which is capable of decomposing under fracture conditions and ensures formation of cavities and permeable channels inside the cement.

Patent applications US2006/0162926, US2006/0166834 describe a hydraulic fracturing method based on the use of a new type of propping particles, as well as the composition of a new material for making gravel packs, based on the use of hydratable cement particles whose average size ranges from about 5 micrometers to about 2.5 centimeters.

The goal of disclosed invention is a higher efficiency of the fracturing treatment for the pay zone.

The goals of disclosed technical solution is application of a new curable proppant (Fig. 1) which exhibits a high chemical stability in regard of the fracturing fluid and the gel breaker; the solution also includes the method of preventing the hydraulic fracture closure and proppant flowback control.

The technical results of the invention accomplishment are the better compatibility of the resin coating with the fracturing fluid and prevention of degradation of the gel breaker and the resin coating. This result is unchangeable viscosity of polymeric-base fluid during the whole fracturing treatment, prevention of untimely settling of proppant in a fracture, better strength of

consolidated proppant pack and improved fracture cleanup from the residual of polymer-based gel. All these factors reduce the proppant flowback at the cleaning stage, well completion and production stages. This invention ensures on the production stage a high initial permeability of proppant packing and long-run steady operation of the fractured well.

The formulated technical results is achieved by the following actions: the resin coating 2 of proppant substrate 1 is coated with at least one additional coating 3 (Fig. 1), which has protective functions by partial or complete prevention of by-side chemical reactions of the resin coating with chemical components of the fracturing fluid and polymer breaker.

The developed method consists of creation of a protective coating (or layer) on the outer surface of the resin coating, preferably, on the basis of polymers, surfactants, waxes, paraffin and mixtures thereof.

The advantages of this approach are listed below.

1) The developed approach makes the production of resin-coated proppants cheaper and simpler in comparison with regular approaches, when additional coating are made from cured resin, e.g., described in patents US5837656, US5955144, US7135231, US7153575.

2) The resin-coated proppant with additional protective coating does not interact with the fracturing fluid during fracturing treatment and even after closing of the fracture. It does not damage the rheology properties of the fracturing fluid and its carrying efficiency. This improved the procedure of the proppant pumping, decrease the probability of untimely stop of treatment job (stop-proppant) and ensures more uniform distribution of the proppant over the fracture height.

3) The resin coated proppant with additional protective coating is indifferent to chemical components of the gel breaker. This improves



performance of gel breakers, provides a better fracture cleanup from the residual gel and, by this, improves the fracture conductivity and reservoir recovery.

4) The listed factors 2 and 3 provide the sustainable quality of the resin coating. The curable coating has a higher strength than that described in patents US5218038, US4585064, US4717594.

5) The extra protective coating 3 can be dissolved in water, oil-bearing fluids, and condensate. Therefore, this external layer does not reduce the permeability of a proppant packing, as it happened for proppants with the protective coating made of cured resin (US4585064, US 4717594).

The method of fracturing treatment of a pay zone was developed; according to this method, the resin-coated proppant is pumped into the fracture, wherein this proppant has at least one additional layer with protective function. This layer prevents completely or partially the reaction of the resin with chemical components of the fracturing fluid and gel breaker.

The production method is developed for curable resin-coated proppant, when at least one protective coating 3 is deposited on the external surface of the resin-coated proppant.

The protective coating 3 comprises partially and/or completely water-soluble or/and organics-soluble and/or degradable compounds, preferably, polymers, surfactants, waxes, paraffin and mixtures thereof.

The protective coating 3 comprises the materials insoluble under downhole conditions.

The possible substances for protective coating 3 are polyolefines, polysaccharides, polylactides, polyglycoles, polyacids, polyacrylamides, polyamino acids, fluoro-polymers, polyacrilates, polyamides, polyvinyls, polyimides, polyuretanes, polycarbonates, polysulfones, polyesters, waxes, paraffins, surfactants, and mixtures thereof, except the curable resins.

Inorganic compounds can be used for depositing of the protective coating 3.

The thickness of protective coating 3 ranges from 10 nm to 1 mm.

The resin coating 2 can be made of compounds suitable for partial and/or complete curing under bottom-hole conditions.

The resin coating 2 may consists from at least one layer.

The resin coating 2 can be a sequence of two and more alternating sub-coatings consisting of cured or curable resin sub-coatings.

As the material for substrate 1, all traditional types of proppants can be used, including those with spherical, elliptical, elongated, angular shapes, with the minimal size in the interval of 1-100 mesh.

Substrate 1 can be made of sand, ceramic, polymer, composite materials, metal, glass and combinations thereof.

Substrate 1 can be made of wood materials.

The protective coating 3 can be deposited on the resin coating 2 by submersion of proppants into the material of protective layer dissolved in appropriate solution (dipping method) and later drying of the proppant under conditions (duration and temperature) that avoid complete curing of the resin coating 2.

The protective coating 3 can be deposited on the resin coating 2 by mechanic-activation method, i.e., mechanical treatment of proppant by fine powder of the protective material in a ball mill.

The protective coating 3 can be deposited on the resin coating 2 by gas-dynamic method, when the protective material is introduced into an air stream that hits the target surface. The air stream temperature is adjusted at the level below the complete curing of the resin coating 2.

The protective coating 3 can be deposited on the resin coating 2 in the

medium of supercritical carbon dioxide fluid. The certain advantages of this approach making of ultra thin, uniform layer with a low roughness. The method helps in optimization of dynamics of the protective layer deposition (preferably polymer); there is no liquid phase at the atmospheric pressure, so no effect of molecules rearrangement due to surface tension forces; the absence of liquid phase for CO<sub>2</sub> dissolvent solves the problem of the residual solvent in the thin surface films; most of potential materials for protective coating 3 are dissolved in the supercritical carbon dioxide. Another advantage is avoiding of curing of the resin coating 2 under the chosen conditions of depositing the protective coating 3.

The protective coating 3 can be deposited on the resin coating 2 using the liquid solutions of tetrafluoroethelene telomers. The essence of the method is dissolving of gaseous tetrafluoroethelene in appropriate solvent and depositing of the solution on the proppant with further radioactive treatment with gamma-rays. The removal of solvent and drying of proppant is accompanied by formation of a solid film with high-quality adhesion to the surface.

The protective coating 3 can be deposited on the resin coating 2 by method of gas-phase surface polymerization from cyclic di-*n*-xylelene. The method uses the fact that in vacuum the molecules of p-cyclophane (and its derivatives) passes through the pyrolysis zone (~600°C) and transforms into active intermediate agent that is condensed on a cold substrate (resin coated proppant). The detail description of the method is given in publication Gorham W.F., J. Polymer Sci., A-1, 1966. V. 4. No.12. P. 3027. By varying the parameters of sublimation, pyrolysis, evaporation, polymerization, one can control the thickness and composition of the protective coating 3.

Under the impact of the reservoir temperature and pressure, the resin coating on the proppant is cured and this creates a uniform and strong permeable mass that prevents the complete closure of the fracture and prevents the proppant

flowback. The protective coating 3 does not prevent coupling of adjacent proppant granules while the curing occurs.

In the disclosed method, the curable proppant can be used on the whole interval of fracturing job or only at the final stage.

The industrial applicability of the disclosed method is proven in experiments that tested the effect of fracturing fluid and gel breaker on Unconfined Compressive Strength (UCS) of resin coated proppants, and the compatibility of the proppant with a gel breaker was tested. Two samples of resin coated proppant were tested.

The sample 1 (reference sample) is a market available resin coated proppant with one curable resin coating. The mass of the resin coating (loss of ignition test data) was 3.8 wt. %. The proppant particle size is 16/20 mesh.

The sample 2 is the same as the reference sample, but with a protective layer deposited by dipping method. The protective layer consists of a mixture of low-pressure polyethylene and paraffin in proportion 7:3. The protective coating thickness was  $20 \pm 5$   $\mu\text{m}$ .

Experiment 1: determine pack strength of curable resin coated proppant in the presence of fracturing gel and polymer breaker.

The sample of 100 g of the proppant (samples 1 and 2) were put into a 500 ml plastic beaker with 100 ml of the guar base linear gel (5 g/L) and 0.1 g of ammonium persulfate (gel breaker), and then the proppant was mixed thoroughly by the mechanical stirrer at room temperature during 1 minutes at the rate of 500 RPM. Then the gel was cross-linked with the use of alkaline solution of boric acid and was vigorously mixed for another 30 sec. The cross-linked gel pH was 12.3. This suspension was put into special steel made curing cell. Generally this is a metal cylinder 38 mm ID with well made-to-measured pistons. To provide a uniform stress condition at the proppant pack ends during UCS measurements,

piston surfaces was parallel, and vertical deviation was not exceed 0.05 mm. The curing cell was equipped with heating jacket. Pistons were equipped with channels for fluid and sealing rings. Safety valve or back pressure regulator with manometer and valve was installed on the cell. Back pressure was 250 psi. Curing cell was equipped with three thermocouples. Two thermocouples control sample temperature and another one controls heating coil temperature. The temperature in the curing cell was kept with the accuracy of  $\pm 1^{\circ}\text{C}$ . The resin-coated proppant was cured under the pressure of 6895 kPa and the temperature of  $100^{\circ}\text{C}$ . The curing time was 1 hour. During the curing period, the temperature and pressure was kept constant for the proppant packing. The accuracy of pressure sustaining was  $\pm 5\%$ . The produced samples were dried in air at room temperature for 24 hours. The proppant packing strength was measured at the “Instron” instrument according to the standard GOST 21153.2-84, ASTM D 3148-02.

Our UCS tests demonstrated that the proppant packing strength of sample 2 was  $170 \pm 10\%$  of that for the reference sample 1. This result demonstrates that the fracturing fluid and polymer breaker has no impact on the quality of the resin coating with a protective coating available.

Experiment 2: test on compatibility of the resin coated proppant with the polymer gel breaker.

The sample of 100 g of the proppant (samples 1 and 2) were put into a 500 ml plastic beaker with 100 ml of distilled water that contains 0.1 g of ammonium persulfate (gel breaker). The samples 1 and 2 are mixed thoroughly with an electromechanical mixer at room temperature for 4 hours. Every 30 minutes, the samples were taken for testing of the ammonium persulfate concentration. The gel breaker concentration was measured by UV-Vis spectroscopy as described in literature (Lo S., Miller M.J., Li J., “Encapsulated breaker release at hydrostatic pressure and elevated temperatures”, SPE 77744). The experiment temperature

was chosen to exclude the thermal decomposition of ammonium persulfate.

The experiment demonstrated that after 4 hours of exposition, the content of ammonium persulfate in samples 1 and 2 was 0.015 and 0.097 %, correspondingly. In other words, the standard market available proppant 1 consumed 84.5 % of the gel breaker. Contrast to this, the resin coated proppant with additional protective coating almost indifferent to ammonium persulfate.

What we claim is:

1. Particles of resin coated proppant comprising proppant substrate and resin coating, wherein the resin surface is additionally covered with, at least, one additional protective coating, which performs protective functions by, at least, partial prevention of by-side reactions between the resin coating and chemical components of the fracturing fluids and polymer breakers.

2. Particles of proppant of claim 1, wherein the material for additional coating is composed from materials including at least partially water and/or organics dissolvable and/or degradable in subterranean conditions incapable to curing polymers, surfactants, paraffin, wax, inorganic compounds and mixtures of thereof.

3. Particles of proppant of claim 1, wherein the material for additional coating is composed from, at least, insoluble in subterranean condition materials.

4. Particles of proppant of claim 1, wherein the material for additional coating is composed from materials including at least partially water and/or organics dissolvable and/or degradable in subterranean conditions crystalline and/or semi-crystalline or/and amorphous polymers and mixtures of thereof.

5. Particles of proppant of claim 4, wherein the additional coating is selected from the group including polyolefins, polysaccharides, polylactides, polyglycoles, polyacids, polyacrylamides, polyamino acids, fluoro-polymers, polyacrilates, polyamides, polyvinyls, polyimides, polyuretanes, polycarbonates, polysulfones, polyesters, waxes, paraffins, surfactants, and mixtures of thereof.

6. Particles of proppant of claim 1, wherein the additional coating is made of inorganic compounds.

7. Particles of proppant of claim 1, wherein the material for additional coating is composed from materials including at least partially water and/or

organics dissolvable and/or degradable in subterranean conditions elastomeric materials with density greater than  $1.0 \text{ g/cm}^3$ .

8. Particles of proppant of claim 1, wherein the thickness of additional coating was from 10 nm to 1 mm.

9. Particles of proppant of claim 1, wherein the resin coating is made from materials that capable to, at least, partial curing under bottomhole conditions.

10. Particles of proppant of claim 1, wherein the resin coating is cured resin.

11. Particles of proppant of claim 1, wherein the resin coating comprises, at least, one layer.

12. Particles of proppant of claim 1, wherein the resin coating is a sequence of two or more sub-coatings made from cured and curable materials.

13. Particles of proppant of claim 1, wherein the substrate is presented by known types of proppants with the minimal linear size in the interval 1-100 mesh.

14. Particles of proppant of claim 13, wherein the substrate has spherical, elliptical, angular or elongated shape.

15. Particles of proppant of claim 1, wherein the material for substrate is sand, ceramic, polymer, composite materials, metal, glass, and combinations thereof.

16. Particles of proppant of claim 1, wherein the material for substrate is wood material.

17. The method of reservoir fracturing that consists of pumping of resin coated proppant into the fracture, wherein the resin coated proppant has at least one additional protective coating which protects, at least partially, from by-side reaction of the resin coating with chemical components of fracturing fluids and gel breakers.



1/1

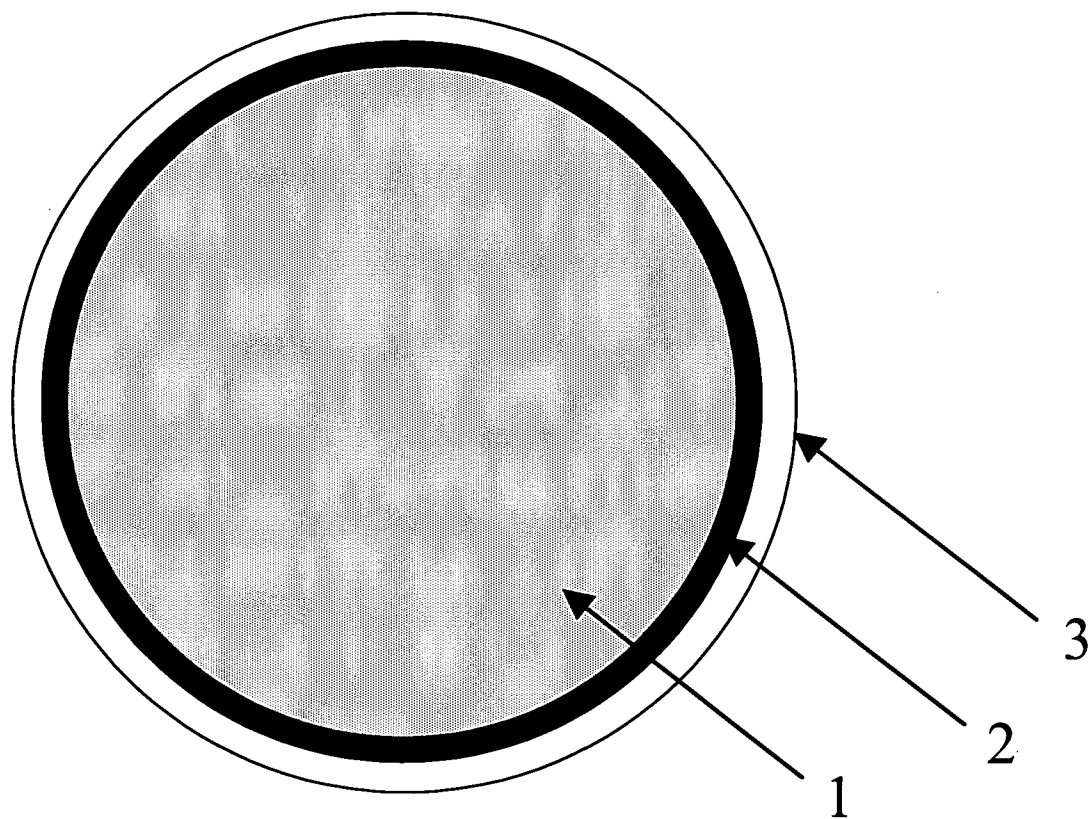


Fig. 1

# INTERNATIONAL SEARCH REPORT

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## A. CLASSIFICATION OF SUBJECT MATTER

**C09K 8/80 (2006.01)**  
**E21B 43/267 (2006.01)**

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)

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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 practicable, search terms used)

RUPAT, RUPAT\_OLD, RUABRU, USPTO, EAPATIS, PAJ, PCT, Esp@cenet

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2005/0194136 A1 (PHILIP D. NGUYEN et al.) 08.09.2005, claims, paragraph [0011]-[0013], [0024]-[0032]	1-11, 13-17
X	US 5837656 A (SANTROL, INC. et al.) 17.11.1998, claims, col. 3, line 49 - col. 5, line 6	1-3, 7-17
X	US 2003/0224165 A1 (ROBERT WILLIAM ANDERSON et al.) 04.12.2003, abstract, claims, paragraph [0053], [0054]	1-3, 7-17

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

☐ See patent family annex.

\* Special categories of cited documents:

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