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MULTIPURPOSE USAGE OF MAGNETIC PROPPANTS DURING SHALE GAS EXPLOITATION

WIELOFUNKCYJNE ZASTOSOWANIE PROPPANTÓW MAGNETYCZNYCH W TRAKCIE EKSPLOATACJI GAZU ŁUPKOWEGO

Abstract: Magnetic material may be added to proppant, as the magnetic marker allows to determine the range and efficiency of hydraulic fracturing. However, magnetic proppant may be also used in flowback fluid treatment and monitoring of environmental pollution. As a result of shale gas hydraulic fracturing, large volume of flowback fluid is created. Flow back fluid have similar properties to fracturing fluid, but it is potentially enriched with large amount of salts and organic compounds leached from shale. Magnetic proppant may serve as a heterogeneous catalyst during organic pollutants decomposition. Additionally, in case of leakage and consequently the fracturing fluid pollution, magnetic proppant is placed into the soil environment. It can be detected using magnetometric methods. This article discusses the above-mentioned issues based on the knowledge and experience of the authors and the literature review.

Keywords: magnetic proppant, shale gas, heterogeneous catalysis, hydraulic fracturing, wastewater treatment, magnetometry

Shale gas

As a result of industrial development, energy consumption is constantly growing. Because of that, ongoing search for new energy sources. Among of them, shale gas is mentioned as a potentially promising. Large deposits of this resource are located in USA and China [1] and they are the global pioneers in the development of shale gas extraction technologies. The shale gas industrial production process started about 30 years ago [2, 3]. Due to the low permeability of shale, from the economic point of view gas flow is insufficient. Acquisition of gas from the rock with low permeability requires its perforation. Therefore, initially shale gas was produced from shale with natural cracks. In order to create artificial fractures hydraulic fracturing technology has been developed.

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Hydraulic fracturing

It is a technological process, aimed at increasing the efficiency of the well. This process is carried out by pumping a fracturing fluid (mixture of water with chemical additives and sand) into the wellbore, under high pressure to produce small cracks - fractures in the rocks. Each fracturing fluid have different composition, according to supplier and geological conditions, but the most important components occurs in all fluids. Hydraulic fracturing fluids are based on water (95-99 %). Proppant, sand or other ceramic material (0.5-2 %) is used to prevent the closure of created fractures, due to rock pressure. Proppant has to have adequate mechanical strength, increasing with the depth of shale. Chemical additives (0.5-5 %) used in fracturing fluid improve the fracturing process. Additives are used: to prevent the swelling of clays (e.g. diethylamine hydrochloride, sodium or potassium chloride), to prevent corrosion of pipes in a wellbore (isopropanol, methanol, chlorobenzene), to prevent stone settling (polyethylene glycol), to prevent precipitation of metal oxides (citric acid), allowing formation of a suspension of sand in water (guar gum, hemicellulose), allowing the subsequent breakdown of gelling agents, responsible for forming a suspension of sand in water (ammonium persulfate, hydrogen peroxide), for maintaining a neutral pH, for the proper operation of gelling agents (potassium carbonate), cleaning and disinfecting borehole (glutaral aldehyde, ammonium chloride), maintaining the proper viscosity of the liquid, with increasing temperature (borate salts, isopropanol), for reducing friction (petroleum distillates), acids (hydrochloric acid) [4]. Many of above mentioned compounds are characterized by a considerable toxicity [5-8]. Proper selection of hydraulic fracturing parameters may be crucial for the economic viability [9]. What is more, hydraulic fracturing awakens numerous controversies. The most important issues are risk of soil and water pollution and huge water consumption [10, 11]. Shale gas exploitation is process, transferring natural environment into heavy industrial zone [12-16]. Because of that, research on alternative method for hydraulic fracturing are developed [16-18].

Magnetic proppants

The decisive factor for the economic viability of hydraulic fracturing is to obtain maximal efficiency of fractures creation [19]. The higher range and amount of fractures created, the higher would be the amount of extracted gas. There are many geological methods of deposits range mapping and data analysis, but all of this methods are expensive and hard to apply in harsh hydraulic fracturing conditions. Because of that idea of cheap and easy-to-detect marker has been developed. This requirements could be possibly met by magnetic marker, substance, that is active in magnetic (natural or inducted) field [20-24].

Two options of introducing magnetic marker to hydraulic fracturing fluid are considered. The first option is to use magnetically active hydraulic fracturing fluid - the whole volume of fluid is then a magnetic marker. This is possible if ferrofluid is in use. But cost of ferrofluid is far too high and magnetic properties of ferrofluid in hydraulic fracturing geological conditions (pressure, temperature) could be significantly decreased. What is the most important for ferrofluid usage, it could be possible to assess where the ferrofluid is, but not where the fractures are. Because of that, nowadays, magnetic marker seems to be one of the components of proppant. For this purpose, feedstock magnetic materials, ferrites and nanomaterials could be considered. Feedstock magnetic materials could be steel shoot or magnetite. They are cheap and available, but their mechanical properties are too weak. Properties of magnetite, because of the different content of impurities are instable. Though, ferrites, the artificial substitute of magnetite could be used. Ferrites, especially MnZn and NiZn ones, have strong, stable magnetic properties. They are widely used in electronics. They are cheap and available. Nano-materials have clearly superior magnetic properties, but their extremely high price [25] exclude them from the possibility of using at the current magnetic materials technology development stage [26].

Magnetic proppants for hydraulic fracturing have already been developed. Magnetic materials added to proppants are NiZn and MnZn ferrites. That allows for obtaining strong magnetic properties. Proppants' magnetic susceptibility is up to $9.22 \cdot 10^{-4}$ m^3kg^{-1} [27], which should allow their successful application.

Proppants' magnetic properties use for soil pollution determination

Magnetic properties of the proppant can be used not only to assess hydraulic fracturing efficiency. It could also be used to assess and detect any fracturing fluid leakage. In case of leakage and consequently the fracturing fluid pollution, magnetic markers are placed into the soil environment. The presence of pollutants in soil can be detected using magnetometric methods [16]. Magnetometry is a surface, non-invasive geophysical method in which the object of measurement is magnetic susceptibility [28-31]. Magnetometric methods allows contaminated soil spatial distribution assessment and immediate *in situ* or *ex situ* soils' remediation.

Hydraulic fracturing flow back fluid treatment

The fracturing fluid, after the fracturing process is pumped from the well. Hydraulic fracturing flow back fluid (HFFBF) have slightly different chemical composition and lower volume compared to the fracturing fluid [10]. Chemical composition change is due to the partial consumption of additives in fracturing process, leaving and crushing of proppant in shale and draining salty underground water from the well. Salinity could possibly be even over 100 $kg \cdot m^{-3}$. Flow back fluid may contain also significant amount of petrochemical hydrocarbons. What is more HFFBF contains some amount of proppant, that is partially crushed as a result of fractures closing.

HFFBF could be treated with membrane processes [32-38], adsorption [39], coagulation [36, 39], electrocoagulation [40, 41], electrodialysis [42], oxidation and Advanced Oxidation Processes (AOP) [43-47], photocatalysis [48]. An alternative for physical and chemical treatment could be biological treatment such as rhizoremediation or algal bioreactors [10], biologically active filtration [49], microbial capacitive desalination cell [50]. HFFBF, oil and gas produced water treatment options are also summarized in some review articles [51-53].

Because of high volume of created HFFBF, low efficiency of biological treatment and unacceptably high cost of membrane treatment, there is still a need for alternative treatment option.

Magnetic marker use for HFFBF treatment

Iron and its compounds, are low cost materials widely used in wastewater treatment. In HFFBF iron and iron based compounds will be present. Fe^{2+} and Fe^{3+} ions source will be ground water pumped back with fracturing fluid. What is more crushed and not-crushed solid proppant will be source of ferrites which area component of ceramic matrix. Because of that two Fe-related treatment mechanisms may be used.

First one is heterogeneous catalysis, occurring on the surface of solid' proppant particles. Numerous processes then takes place, including: oxidation and reduction of pollutants and catalyst, precipitation and co-precipitation of metal oxides and hydroxides, adsorption and coagulation. Additionally, as a result Fe^{2+} and Fe^{3+} ions could be transferred to aqueous phase. Examples of heterogeneous catalytic reactions are shown in Figure 1.

Second treatment mechanism is homogenous catalysis, related with presence of dissolved Fe^{2+} and Fe^{3+} ions in aqueous solution, that start Fenton/pseudo-Fenton reaction [54] in a presence of oxidant, such as H_2O_2. The idea of Fenton/pseudo-Fenton reaction is effective production of free radicals that oxidized organic pollutants. First equation (1) is called Fenton reaction shows the oxidation of Fe^{2+} to Fe^{3+} ions additionally resulting in decomposition of H_2O_2 into $^{\bullet}OH$ radical. If Fe^{2+} ions are replaced with Fe^{3+}, reaction is called pseudoFenton reaction. Furthermore, many other reactions (2)-(5) occurs [55]:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + {}^{\bullet}OH \tag{1}$$

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + {}^{\bullet}O_2H + H^+ \tag{2}$$

$$Fe^{2+} + {}^{\bullet}OH \rightarrow Fe^{3+} + OH^- \tag{3}$$

$$Fe^{2+} + {}^{\bullet}O_2H \rightarrow Fe^{3+} + OH^-_2 \tag{4}$$

$$Fe^{3+} + {}^{\bullet}O_2H \rightarrow Fe^{2+} + O_2 + H^+ \tag{5}$$

Reactions (1)-(5) may also occurs on solid catalyst surface. It was confirmed, for solid metallic iron, Fe^0 (zero-valent iron, ZVI) catalyst in treatment process including simultaneous usage of Fe^0 and hydrogen peroxide, H_2O_2, called ZVI/H_2O_2 or Fe^0/H_2O_2 process [47], that it is highly effective in HFFBF treatment. Hydroxyl radicals may be produced not only with metallic iron Fe^0 as an catalyst. Any other metal cations could be used (reaction (6)) for replacement of Fe^{2+} ions (reaction (1)):

$$M^{n+} + H_2O_2 \rightarrow M^{(n+1)+} + OH^- + {}^{\bullet}OH \tag{6}$$

It could be especially important if MnZn and NiZn ferrites are in use as a part of magnetic proppant. Ferrites from the chemical point of view, belongs to the spinel group. Spinels are compounds of the general formula AB_2O_4, where A can be a metal in the second oxidation state and B can be a metal in the third oxidation state. In MnZn and NiZn ferrites, Zn, Ni and Mn are in second, while the Fe is in third oxidation state. General ferrite formula is $(Mn/Ni)_xZn_yFe_2O_4$, $x + y = 1$.

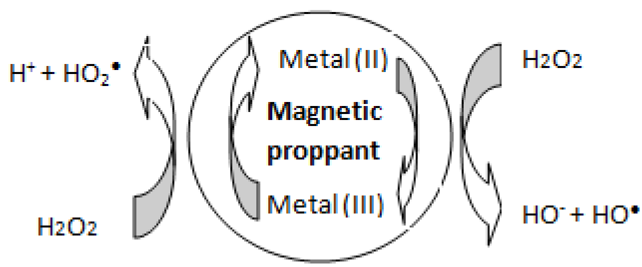

Fig. 1. Examples of reactions takes place on solid catalysts' surface

What is more, hydroxyl radicals could react with dissolved ions, producing other radicals (reactions (7), (8)), such as carbonate or sulfate radical anion:

$$^{\bullet}OH + CO_3^{2-} \rightarrow OH^- + CO_3^{-\bullet} \quad (7)$$

$$^{\bullet}OH + SO_4^{2-} \rightarrow OH^- + SO_4^{-\bullet} \quad (8)$$

In classical Fenton/pseudo-Fenton reaction, source of hydroxyl radical ($^{\bullet}OH$) creation is hydrogen peroxide (reaction (1)). Then, it may be converted into other radicals. But some process modifications are known, in which other oxidants, such as persulfates ($S_2O_8^{2-}$), are source of sulfate radical anions ($SO_4^{-\bullet}$), as it is shown in reaction (9). It is especially important, as in the composition of fracturing fluid, oxidants such as ammonium persulfate may be present, what allows for Fenton/pseudo-Fenton reaction enhancement

$$S_2O_8^{2-} \rightarrow 2\ SO_4^{-\bullet} \quad (9)$$

Created radicals, reacts with organic pollutants causing their oxidation, according to reactions (10)-(15):

$$^{\bullet}OH + RH \rightarrow H_2O + R^{\bullet} \quad (10)$$

$$R^{\bullet} + O_2 \rightarrow ROO^{\bullet} \quad (11)$$

$$R^{\bullet} + Fe^{2+} \rightarrow R^{+} + Fe^{3+} \quad (12)$$

$$ROO^{\bullet} + RH \rightarrow ROOH + R^{\bullet} \quad (13)$$

$$ROOH + Fe^{2+} \rightarrow RO^{\bullet} + Fe^{3+} + OH^- \quad (14)$$

$$ROOH + Fe^{3+} \rightarrow ROO^{\bullet} + Fe^{2+} + H^+ \quad (15)$$

One of requirements for Fenton homogeneous reaction are acidic conditions, usually pH close to 3.0 is considered. In case of HFFBF pH is higher, about 6.0. Because of that homogenous reaction efficiency might be decreasing, as a result of radical scavenging and iron hydroxides precipitation/coagulation. In higher pH efficiency of process is usually lower. On the other hand, there are some reports of the Fenton reaction effective use, even under in high pH [56]. However, as the hydraulic fracturing is periodic process, HFFBF is generated periodically and then collected in tanks. There is no need for high rated process, reaction may be slow, which greatly increases applicability of heterogeneous catalysis with magnetic proppant. For increasing reaction efficiency additional oxidant, such as hydrogen peroxide, ammonium persulfate or other, may be added to increase effect generated by residual oxidant, that was initial compound of fracturing fluid. As a result of oxidation process, organic pollutants could be possibly oxidized to carbon dioxide and water, but it is hard to obtain complete mineralization. Usually, only partial oxidation, to low-molecular-weight organic compounds, occurs. As a result, much more polar compounds are created. They are generally less toxic and more biodegradable than the parent compounds. It was proved, that magnetite catalyst allows not only for organic pollutants removal [57, 58], but it is also useful for heavy metals and metallic ions removal, such as for example molybdates [59] or dichromates(VI) [60] and others, that could be possibly leached from the wellbore.

As a result application of magnetic proppant for HFFBF treatment, should allow the removal of organic contaminants and heavy metals to acceptable levels and then discharge treated HFFBF to the receiver. Magnetic proppant could be easily separated from the HFFBF using electromagnetic methods.

Conclusions

Magnetic proppants, may act as the magnetic marker during the hydraulic fracturing process, allowing to determine efficiency of fracturing. Magnetic proppant may additionally be used, during hydraulic fracturing flow back fluid treatment, as the organic compounds' decomposition catalyst. Using heterogeneous and homogenous Fenton/pseudo-Fenton catalytic mechanisms, amount of organic pollutants contained in flow back fluid could be possibly decreased do acceptable level.

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