



Hydration induced morphological change on proppant surfaces employing a calcium-silicate cement system



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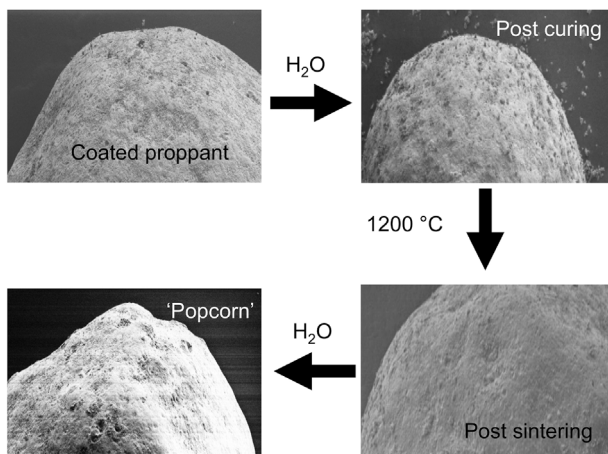
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GRAPHICAL ABSTRACT



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ABSTRACT

Commercial aluminosilicate proppant particles have been coated with Ca-Si oxides, with the aim to provide an in-situ increase in the angularity (decrease in Krumbein roundness value) to facilitate their immobilization. Ca-Si oxide systems have been synthesized via sol-gel, cured, and sintered at 1200 °C using (a) CaCO₃, (b) CaCO₃ + orthosilicic acid (Si(OH)₄, SA), and (c) CaCO₃ + fused silica (SiO₂, FS). When the proppant is cured in the presence of CaCO₃ and silicic acid the coatings undergo a significant compositional change, while sintering results in the conversion of the cured samples to ceramic agglomerates with the desired “popcorn” shapes. The best results are obtained in the presence of Si reagents, and hydration of these sintered proppants allows for a distinct increase in the angularity, which is the desired transformation to allow the proppant to be locked-in-place once located in the reservoir. The samples have been characterized at each stages of preparation by

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scanning electron microscopy (SEM) with associated energy dispersive X-ray spectroscopy (EDX), X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD) and infrared (IR) spectroscopy.

1. Introduction

With a reserve estimated at 482 trillion cubic feet (Tcf) of hydrocarbon, shale gas has the potential to be the primary energy source for power generation in the US for the coming decades [1]. The ability to extract shale oil and gas economically has been enabled by the development of hydraulic fracturing along with horizontal drilling techniques [2]. Hydraulic fracturing (also known as “fracing” or “fracking”) uses water, a proppant, and various chemical additives pumped at high pressures into the well bore, to induce fracturing of the impermeable shale source rock, creating sufficient permeability for the gas to migrate into the well bore and from there to the surface [3–5]. During this process a proppant material is used to “prop” open the fractures, stabilizing the well and allowing the gas to escape once the pressure is removed. These proppant materials need to be sufficiently hard to withstand the stress upon removal of the pressurized fluid. In addition, it is desirable that they are of uniform size and shape to enhance the flux of the oil, natural gas and natural gas liquids out of the rock formations [6].

Most proppant consists of sand, made from high purity sandstone; however, alternative ceramic particles made from sintered bauxite are used [7]. The proppant must be mobile enough in the fluid to be introduced into the fractures in the rocks during the pumping of high-pressure water based fluid in the stimulation process. However, once in place the proppant should not be mobile. Unfortunately, the proppant can unintentionally shift from the desired location in the well formation, and the subsequent loss of proppant is a leading cause of production decline. Counter to this situation, if the proppant is insufficiently mobile then only a fraction of the fracture can be propped since the proppant will create a bridge prior to the tip of the fracture. Attempts to address this problem using elongated shapes or other specific non-spherical materials have not been successful since a non-spherical shape is more difficult to pump. What is needed is a proppant that is non-aggregated during pumping, but is then aggregated or immobilized downhole without loss in permeability. To this end several methods have been investigated, including: coating proppant with liquid surface-modification agent (SMA) resins [8], oligomers of furfuryl alcohol resin [9], various adhesive substances [10], organo-silanes [11], thermoplastic resins [12], immobilization via molecular anchors [13,14] and microwave irradiation [15]. An alternative route would be a spherical proppant that once in place “cures” to create a lock-and-key interaction via a morphological change on proppant surfaces (Fig. 1).

We have previously shown that, in the presence of a calcium binding agent, CaCO_3 may undergo a surface reorganization [16]; however, such a process would require the use of additional chemicals, when public pressure is to limit chemicals employed downhole [5,17]. In contrast, Portland cement represents a material that under mild conditions (hydration) undergoes reactions that result in morphological change as well as fusing individual grains together [18]. Furthermore, the hardness of set cement would be suitable for a proppant material once in place [19]. Given that typical ceramic proppants are aluminosilicate then the potential inclusion of a calcium source could produce one or more of the cement main phases: tricalcium silicate ($3\text{CaO}\cdot\text{SiO}_2$), dicalcium silicate ($2\text{CaO}\cdot\text{SiO}_2$), and tricalcium aluminate ($3\text{CaO}\cdot\text{Al}_2\text{O}_3$) [20,21]. Herein, we have investigated the surface reaction of a typical commercial aluminosilicate proppant with CaCO_3 (including at high temperature) and its subsequent hydration to determine if any suitable surface morphological change occurs through reaction with the templating aluminosilicate proppant. The known interaction of fumed silica with cement materials [22] and our prior report [23], which has

shown that the reaction of CaCO_3 with either orthosilicic acid ($\text{Si}(\text{OH})_4$, SA) or fused silica (SiO_2 , FS) can create distinctive nodular morphology under mild conditions, would suggest that a combination of both CaCO_3 with a silica source would offer the ability to react on the surface of the proppant forming a calcium silicate cement (CSC). Such materials have been proposed as fast setting bone cements [24]. A representation of the procedures used herein is shown in Fig. 2.

2. Materials and methods

2.1. Materials

ACS grade calcium carbonate (CaCO_3 , Cal), orthosilicic acid [$\text{Si}(\text{OH})_4$, SA], and fumed silica (SiO_2 , FS) were purchased from Sigma Aldrich and used as received. Deionised (DI) water (Mallinckrodt chemical works). Carbon dioxide was 99.99% pure, Matheson TRIGAS. The proppant (P) was obtained from CARBO Ceramics, Inc., and has a main composition of aluminosilicate, a range of particle size of 60–700 μm (average size = 370 μm), and a density of 1.57 g/cm^3 .

2.2. Sample preparation

A summary of the reagent combinations and the sample abbreviations is given in Table 1. Samples with and without proppant were processed in a near identical manner (except for the presence or absence of proppant), the only difference being the relative water quantities employed. In all the cases solid CaCO_3 (500 mg) was added to deionised water (250 mL) and stirred at ambient pressure under a flow of CO_2 for 1 h (5 h in the case of just CaCO_3 as reagent, i.e., Cal). In the experiments with proppant, 4.0 g of proppant were employed. Where used (Table 1), 50 mg of either orthosilicic acid (SA) or fumed silica (FS) was added, while stirring and bubbling with CO_2 for 4 h. After this time, the reaction was kept under a CO_2 atmosphere with continuous stirring for 20 h. Finally, the samples were filtered by gravity and dried in an oven at 70 $^\circ\text{C}$ for 1 h to be characterized before the curing process. The samples with proppant were sieved (70 mesh = 0.212 mm) after drying, keeping the coated proppant (retained by the sieve) and the excess powder (passed through the sieve) separated. It should be noted that ‘excess powder’ refers to the powder that is formed from the proppant surface due to physical handling of the proppant, in contrast ‘powder’ is used to describe material formed in the absence of proppant.

The curing process was performed by adding distilled water to small amounts of sample and aging for 1 week under ambient indoor conditions (ca. 20 $^\circ\text{C}$, 1 atm). The samples with proppant were cured after having added back a small amount of excess powder to the proppant. After 1 week, most of the water was removed and the samples were dried in an oven at 70 $^\circ\text{C}$ for 1 h. In order to test for the effect of cure time, the sample synthesized using calcium carbonate and orthosilicic

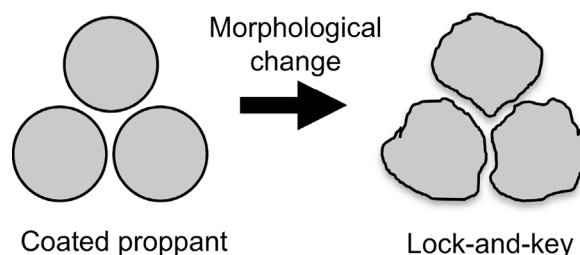


Fig. 1. Schematic representation of a post placement morphological change on proppant surfaces to create an interlock to immobilize the proppant.

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