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Key words: *encapsulated acid system, functionalized nanosilica, digital core analysis, formation damage*

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INTRODUCTION OF A WORKFLOW FOR TOMOGRAPHIC ANALYSIS OF FORMATION STIMULATION USING NOVEL NANO-BASED ENCAPSULATED ACID SYSTEMS

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During the production time, it is crucial to manage the reservoir efficient productivity and keep it at a profitable level. Matrix acidizing in carbonate reservoirs is a common course of action to increase the efficiency of production. The present project is based on an integrated multi-disciplinary plan as an arena to merge traditional and novel technologies in the field of petroleum engineering, petroleum geoscience, chemical engineering, computer vision and mineralogy. Some crucial parameters such as permeability/porosity changes occurred during carbonate acidizing are modelled and analyzed based on various modern technologies, such as, the novel digital rock technologies. A waste variety of nanoparticles is also used in order to design a novel acid mixture for stimulating the carbonate reservoirs. Specifically, this study is considered as a one-step forward in development of smart encapsulated acid systems using a range of hydrophobic silica nanoparticles in various grades of hydrophobicity. Moreover, the present study can be considered as the first practical example for application of digital rock physics in improvement of acidizing operation in Iran and Russia.

The proposed research methods are consist of preparation of encapsulated acids, sample and data collection, conventional core analysis, digital core analysis, lab experiments and modelling and conclusion. Characterization of the efficiency of this process was once more characterized using the aforementioned digital rock technologies to visualize the effect of encapsulated acid fracturing operation, impact of surface modification of silica NPs on the etching efficiency, the physical properties of core samples, and subsequently the final productivity index. Thin section, SEM and FE-SEM analysis was then performed to further evidence the efficiency of this method. Moreover, the efficiency of this method was categorized based on the identified mineralogy and rock composition.

It was concluded that the dissolution rate was significantly increased as a result of acid neutralization control and the reaction rate decreased which in turn resulted in more homogenous patterns of wormholes, higher permeability, and so, more successful acid treatment. Thanks to the reduced accessible surface of acid systems caused by their emulsion-based nature, it was found that this novel encapsulation process can reduce the risks of corrosion in all the equipment in surface and bottom hole. It naturally reduces the extra costs of corrosion-related damages and subsequent workover operations, which are the common need of most of the wells treated by conventional acid fracturing operations.

Key words: encapsulated acid system, functionalized nanosilica, digital core analysis, formation damage

INTRODUCTION

One of the most deteriorating problems occurred in the oil and gas industry, usually during drilling operation, enhanced oil recovery and cementing processes, is reservoir formation damage [1, 2]. This problem, which is frequently caused by migration of dispersed solid particles and colloids through porous media, may significantly reduce the absolute permeability of medium [3]. Regardless of the reason, this phenomenon can mainly lead to the reduction of petroleum production rates or decrease in the injection capability of porous media [4, 5].

While a wide range of stimulation processes is introduced to tackle the formation-damage-based problems, when it comes to carbonate formations, fracturing treatments, including hydraulic fracturing and acid fracturing, have been widely welcomed due to their remarkable results [6, 7]. Creating a couple of fractures inside the pay zones connecting the virgin zones to the wellbore, damaged regions and their deleterious effects on production rates are simply bypassed. Apart from tackling the formation damage, using these methods have exhibited desirable results in some fields else, such as productivity index increase in tight shaly gas reservoirs and enhanced geothermal system (EGS) technology [8, 9].

Despite the proved advantages of hydraulic fracturing, however, there exist some detrimental drawbacks behind this process, such as lower permeability of fractures originated from crushing the proppants as well as proppant settling down near the wellbore [10, 11]. Eliminating such demerits, acid fracturing has gained greater deal of at-

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tention during the last years. Chemical reactions of the acid with fractured rock etch the fracture faces and create conductive pathways for fluid flow, even when the fractures are closed, which in turn eliminates the needs for proppants, and increases the productivity index of reservoir [12]. In terms of carbonates, as the most common reservoir rocks in Middle East, hydrochloric acid (HCl) is the most beneficial acid which can effectively dissolve calcite minerals (also called as "etching") and enhance the fractures' conductivity [13-15].

However, over-reaction of acid in the first distances of injected zones, peculiarly in terms of shaly carbonates, is among the most challenging issues. This extra reaction of acid might lead to bulk dissolution of formation, instead of non-uniform etching, which decreases the fracture conductivity after closure [16, 17]. Besides, it can soften the fracture face more than required and increase the risk of totally fracture closure. Another issue, as mentioned earlier, are the corrosion-related risks in equipment caused by high concentration of acids required for this process [18]. While formic acid, acetic acid, and some of organic acids are also considered as practical candidates in carbonates in case the prevention of metal corrosion brings about challenging problems, their lower dissolving powers, higher costs compared to HCl, and the less understood acid-rock reactions have significantly limited their applications [10, 19, 30].

A pioneering approach, which has recently been introduced, is increasing the stability of acidic colloidal systems using the encapsulation of acid droplets by nanoparticles [22]. Particle-stabilized acidic colloids, synthesized based on hydrophobic NPs, have recently exhibited outstanding features, appropriate stability, and remarkable resistance against coalescence, even in the harsh conditions of reservoirs [23, 24]. In this method, encapsulated acid droplets, which are injected through the fracture openings in the form of powders (acid-in-air emulsions), can be released by changing the pH, injection of de-emulsifiers, or simply decreasing the injection pressure, which cause the fracture closure when the overburden pressure overcomes [25-27; 31; 36-38].

However, the effect of hydrophobicity degree on the performance of acid fracturing is still not well investigated [28]. Besides, due to the lack of in-situ measurement of encapsulated acid fracturing effects in previous attempts, when the fractures have not been in their inherent conditions, obtained results lack the enough accuracy and cannot reveal the actual efficiency of fracture face etching phenomenon [26].

This study aims to fill the gaps by designing nanosilicas, varied in the degree of hydrophobicity, and investigation of their effects on the effectiveness of encapsulated acids in fracturing operation simulated by core-flood systems. Results can be precisely analyzed using the novel digital rock physic techniques, such as micro-computed (Micro-CT) scan, SEM, XRD, etc. and then modeled using design of experiment (DOE) methods. This can significantly enhance the effectiveness of acid fracturing operation and provide a model for prediction of results.

METHOD OF RESEARCH

Synthesis of silica NPs of Various grades of hydrophobicity

To achieve the desired hydrophobicities, surface modification of nanosilica will be primarily performed with 3-glycidoxypropyl-triethoxy silane (we call it GPTS). This process will be performed using ultrasonically vibration of the mixture of bare nanosilica, ethanol solvent, and deionized water for 1 hr. An especial amount of silane coupling agent will be then added to this mixture, while pH value will be adjusted in the range of 3 to 5. The prepared mixture will then be continuously stirred for around 24 hours. Having cooled down the mixture, colloidal solution will be separated using centrifugal forces which is followed by several times washing process by ethanol to eliminate the excessive silane. Modified nano-SiO_2 will be then dried at 80 °C for 24 hours, and, at this step, GPTS-SiO_2, indicating the least amount of hydrophobicity after bare nanosilica, will be obtained. In the next step, in order to increase the hydrophobicity of NPs, double amount of GPTS on nanosilica (DGPTS) will be added on the surface of nanosilica, with the same approach as GPTS-SiO_2. At the end, GPTS-based nano-additives will be modified with propyl silane (PGPTS) to achieve the highest degree of hydrophobicity[29]. As it can be seen in Table 1, the employed procedure in previous researches has resulted in successful alteration of hydrophobicity.

Materials and methods

Materials: Carbonate borehole whole core was collected and used for coring of the required plug sample. The prepared rock sample was analyzed for porosity, permeability, mineralogy, and pore distribution through optical petrography, XRD, XRF, mercury intrusion measurements and μ-CT scan methods. The rock sample used in this study was classified as a wackestone/packstone carbonate with high amount of microporosity showing a dominant contribution to porosity (~80%) comes from intercrystalline micropores in the range of 100nm to 4μm.

We used Ethylene diamine tetra-acetic acid (EDTA) as a stimulating fluid. The EDTA solution and deionized water were prepared in the laboratory. All chemicals used in this study were of analytical grade and were used as received without any further purification.

Core-flood experiments: Core-flood experiments were conducted through injection of the prepared EDTA fluid sample at ambient conditions (See figure 1). Details of the experimental setup and the experiments were described in (Qajar et al., 2013). The chemical dissolution experiment was conducted in a small core plug of 7mm diameter and 17mm length in an effort to acquire μ-CT images at high resolution. The core sample was first vacuum saturated with deionized water. The initial

Table 1: The main chemical and physical properties of the synthesized silica NPs[29]

Name	Drilling fluid*	Type		Type of modification	Zeta potential (mv)	Size** (nm)
		Bare silica	Modified Silica			
SiO_2_1***	-	●		No modification (Bare silica)	-	11
SiO_2_2	NDF-4	●		No modification (Bare silica)	- 51.3	15
SiO_2_3****	-	●		No modification (Bare silica)	-	20
GPTS@ SiO_2_1	NDF-1		●	3-glycidoxypropyl-triethoxy silane (GPTS) on SiO_2_1	-	11
GPTS@ SiO_2_2	NDF-2		●	3-glycidoxypropyl-triethoxy silane (GPTS) on SiO_2_2	- 43.6	15
GPTS@ SiO_2_3	NDF-3		●	3-glycidoxypropyl-triethoxy silane (GPTS) on SiO_2_3	-	20
DGPTS@ SiO_2_2	NDF-5		●	3-glycidoxypropyl-triethoxy silane (GPTS) on SiO22 (double amount compared with GPTS@ SiO_2_2)	- 35.9	15
PGPTS@ SiO_2_2	NDF-6		●	3-glycidoxypropyl-triethoxy silane (GPTS) and propyl silane on SiO_2_2	- 21.3	15

* NDF stands for nano-based drilling fluid

** Measured using the particle size analyzer device (Microtrac Company, MN42x, Japan)

*** Used in preparation of GPTS@SiO_2_1

**** Used in preparation of GPTS@SiO_2_3

porosity of the sample was measured by the saturation method (φ_0=21.1%). The fluids were injected at ambient conditions using a motor-controlled syringe pump and the pressure drop along the core length was monitored by a differential pressure transducer and recorded by a computer.

The initial permeability of the core was first measured by injecting deionized water at different flow rates (k_0=3.15mD). After that, the sample was flushed with EDTA solution at a rate of 8.0×10-10 m^3.s^{-1}. The injection of EDTA was terminated after approximately a twice increase in permeability. Finally, the core was dried in an oven and then imaged again. The measured final porosity of the sample was determined equal to 22.5%.

X-ray μ-CT imaging: Characterization of the efficiency of the performed dissolution process was visualized using the micro-CT imaging technique. Note that micro-CT images before and after the treatment were acquired using the available Australian National University μ-CT facility (Sakellariou et al., 2004). Images were taken with a field of view of at least 8.5×8.5×8.5mm and a resolution of 5-10 micron. The tomograms in each state were composed of 2048^3 voxels.

Image analysis: The image processing steps were previously described in details by Qajar and Arns (2016). One of the most important steps is image registration. As a matter of fact, the pre- and post-dissolution images need 3D voxel-to-voxel image registration before any comparison between the two successive images can be made. We used a 3D registration technique developed by Latham et al. (2008), which allows voxel-precise overlays of tomographic images of successively disturbed specimens.

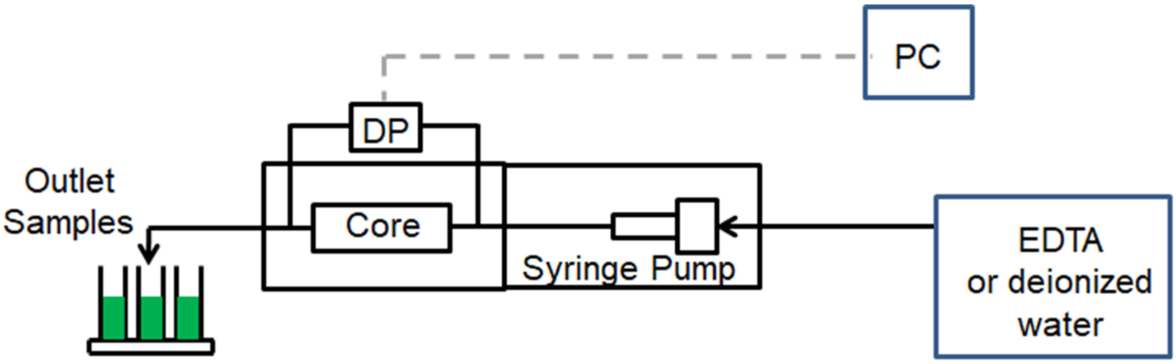

Figure 1: Schematic of experimental setup at ambient conditions

Preparation of encapsulated acids

Synthesized NPs will then be added to hydrochloric acid (HCl) solution at different ratios and will be blended using the mixers at predefined speeds. Designed protocol will end in self-assembling of silica nanoparticles at the air-water interface and formation of acid-in-air emulsions. Encapsulated acid system, in the shape of powder, will be prepared and ready to be used in the following steps of experiments [25, 26]. Characterization of encapsulated acids will be then performed using SEM, particle size analyzer, and optical microscopy techniques.

Pore-scale characterization

Proper outcrops will be prepared based on geological surveys, and required samples will be achieved using coring operation. The physical properties of cores, including their porosity, permeability, mineralogy, pore distribution status, and so on, will then be characterized using XRD, XRF, Micro-CT scan, etc.

Core-flood experiments and digital rock physics

At the next step, core-flood experiments will be conducted using the selected core samples, split in middle simulating the fracture faces, where different the prepared encapsulated acids will be injected inside the simulated fracture opening. Having increased the overburden pressure mimicking the fracture closure conditions, acid droplets will be released and fracture face etching will be triggered.

Characterization of the efficiency of this process will be once more characterized using the aforementioned digital rock technologies to visualize the effect of encapsulated acid fracturing operation, impact of surface modification of silica NPs on the etching efficiency, the physical properties of core samples, and subsequently the final productivity index.

Sample analysis

Three Micro-Plugs are taken form a carbonate reservoir which is located in the Middle East. The Micro-City imaging has been done with the resolution of 5-10 micron. At this stage of the work, the images of the plugs before treatment are available.

RESULTS

Figure 2 illustrates examples of top- and side-view slices through the registered pre- and post-dissolution µ-CT images of the sample. The interested readers can refer to (Qajar and Arns, 2016) for more images and details. The visual observations of the images reveal the reactive fluid was consumed over small parts of the mineral surface area leading to the formation of a few highly conductive flow channels (wormholes) and a small increase of porosity.

The formation of highly conductive wormholes leads to permeability improvement. As a consequence, the productivity index of the well will be increased. The produc-

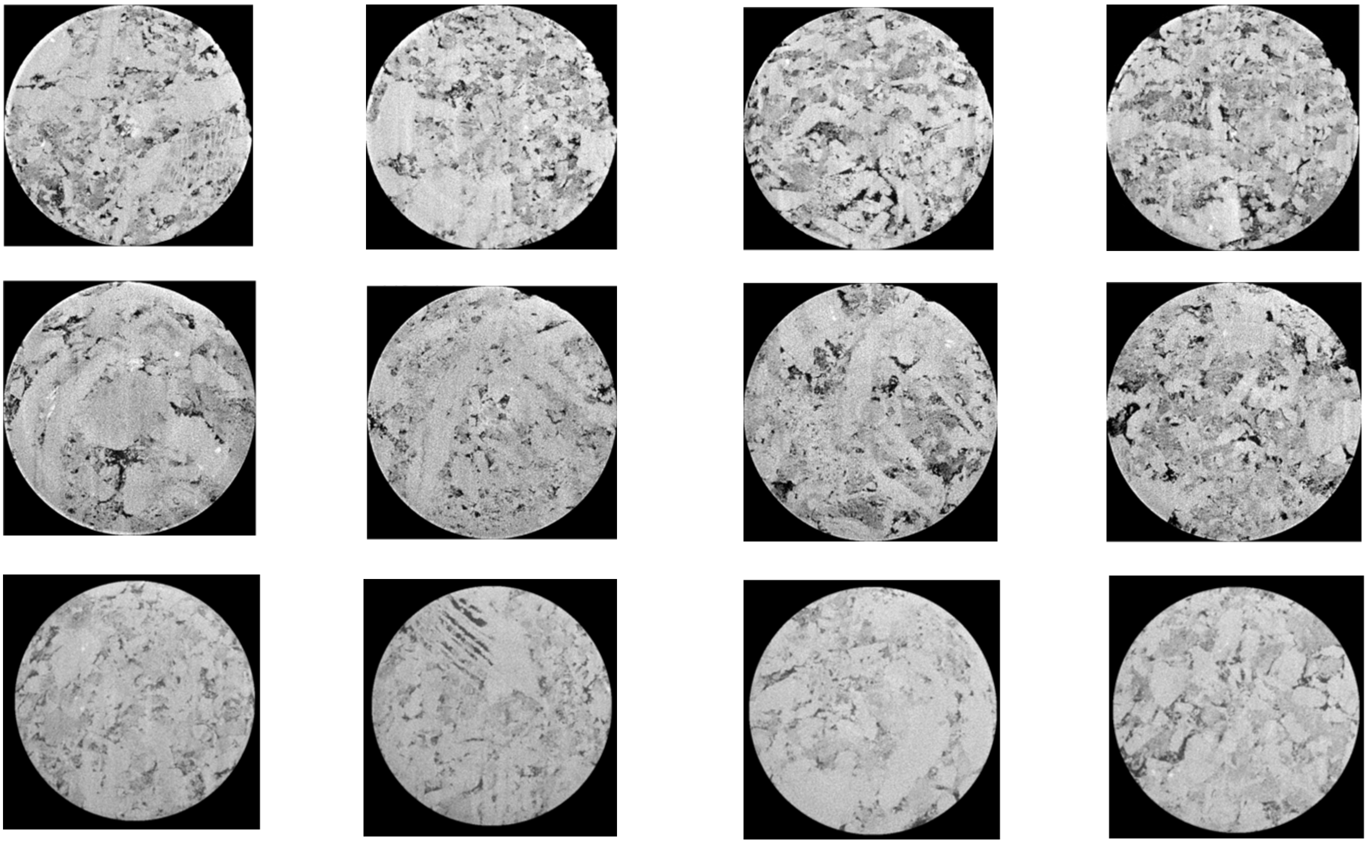

Figure 2: Micro-City imaging of the plugs before treatment

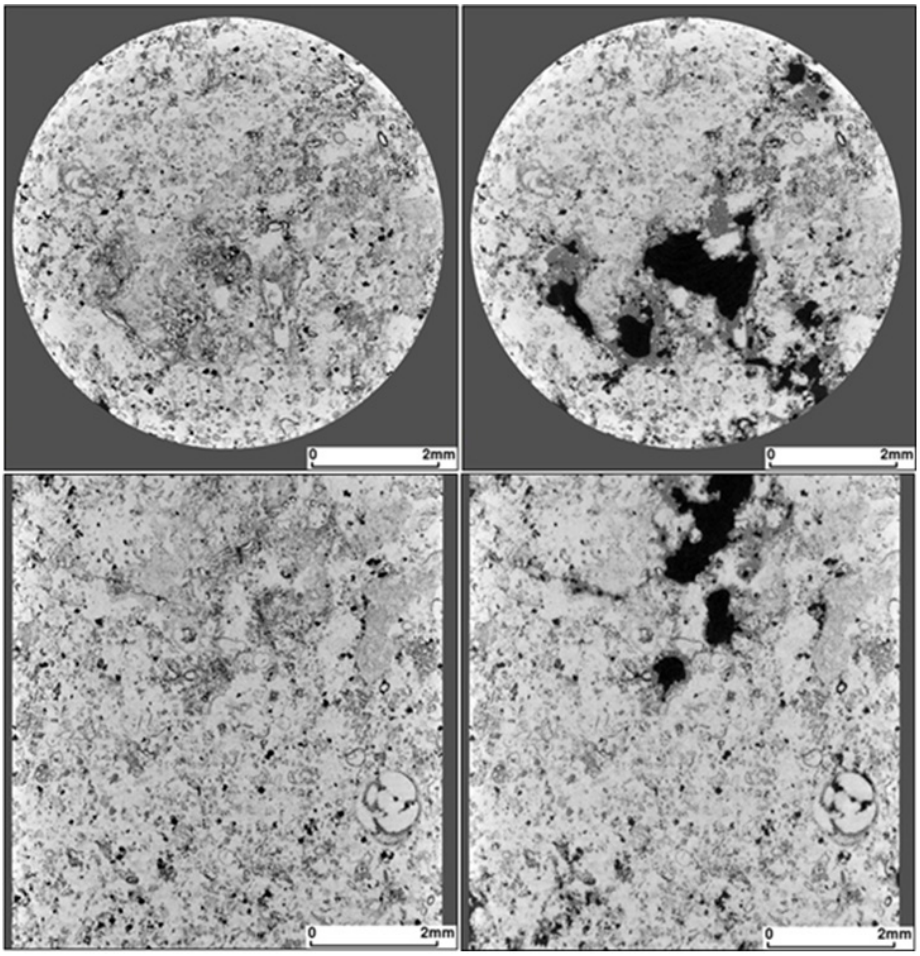

Figure 3: An example of top-view slice (top row) and side-view slice (bottom row) of registered images of the sample before (left) and after (right) the dissolution experiment

tivity index is the ratio of the total liquid surface flowrate to the pressure drawdown at the midpoint of the producing interval, which has a direct relationship with permeability. In other words, the higher the permeability the higher the productivity index. Formation of wormholes with deep penetration also leads to the skin reduction. Based on the results and the μ-CT imaging analysis, it is obvious that EDTA has been working properly as a chelating agent and stimulation fluid. Using EDTA results in a successful acidizing operation by creating deep wormholes, decreasing the reaction rate and increasing the dissolution rate (between rock and stimulation fluid).

In the successful acidizing operation, the wormhole penetration highly enhances and with bypassing the formation damage the skin highly reduces. The permeability enhancement and skin reduction result in the productivity index enhancement and as a result, improves the well potential. The productivity index can be expanded to a semi-steady state Darcy law type formulation where:

$$J = \frac{0.00708kh}{\mu B\left[\ln\frac{r_e}{r_w} - \frac{1}{2} - S\right]} \tag{1}$$

where k is permeability (mD), h is net thickness (ft.), μ is fluid viscosity (cp), B is formation volume factor (rb/STB), r_e is external boundary radius (ft.), r_w is wellbore radius (ft.) and S is the skin.

CONCLUSION

Owing to some laboratory challenges pertinent to acidizing operation, the potential of high-pH calcite-dissolving fluids in carbonate dissolution systems was analyzed in this work through the injection of EDTA, as an appropriate chelating agent and stimulation fluid, inside the selected core samples and analyzing the results based on novel digital rock physics. It was reconfirmed that using the high-pH dissolving fluids can eliminate the need of high pressures required for preventing gaseous CO_2 liberation in typical acidizing operation. It was found that EDTA dissolving operation could cause a significant increase in permeability values, while porosity changes might be negligible (merely around 1.4% increase). Based on the μ-CT imaging results, as one of the first practical applications of digital rock physics in the evaluation of high-pH calcite-dissolving systems, it was found that the obtained results were pertinent to the fact that reactive fluids have been consumed over the small parts of mineral surface area which in turn can cause the formation of some highly conductive wormholes, without significant increase in porosity.

Besides, it was found that reduced pH of proposed dissolving fluid can deal with the risks pertaining to equipment corrosion and reduce the subsequent extra costs, such as those linked with formation damages and workover operations. Furthermore, in field application, the highly conductive cannels created inside the reservoirs can avoid the corresponding lost from the associated reduction in flow rate. In addition, this research is a groundbreaking example to present the advantages of pore-scale analysis, using µ-CT imaging method, in the precise investigation of calcite dissolution operation.

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