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## Research paper Fabrication of kaolin-based cement plug for  $CO<sub>2</sub>$  storage wells

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### article info abstract

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A new material from clay was developed that has a resistance to carbon dioxide flow in upstream for plugging CO2 storage wells. Thermogravimetric and thermal stability of this material showed a minimal variation in weight loss with incremental heating up to 420 °C. Adsorption-desorption of  $CO<sub>2</sub>$  at various modeled temperature showed an exothermic and spontaneous process with maximum adsorption capacity of 775 mg/g obtained at 640 psi (43.5 bar) and 50 °C. Higher pressure led to more storage capacity with physisorption hysteresis curves. Freundlich and BET models best fit the equilibrium adsorption data with average regression coefficient of 0.995. The new material can substitute the conventional cement plugs for upstream carbon sequestration and prevents the migration of the stored CO2 through the cement plug to the surface. Also the developed new cement showed no chemical interaction which confirms the economical impact of using this cement to plug the CO<sub>2</sub> sequestrations wells.

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#### 1. Introduction

The implementation of carbon capture and sequestration in Enhanced oil (EOR) and gas (EGR) recovery has recently gained great interest in terms of recovery cost, feasibility, and environmental remediation. This emerging technology has dual advantages of reducing the emission of  $CO<sub>2</sub>$  into atmosphere and recovering the oil and gas that left after secondary processes. However, maintaining  $CO<sub>2</sub>$  in gas and oil wells is a challenge in  $CO<sub>2</sub>$  sequestration and EOR and EGR recovery. The downhole forces on the pumped cement between the casing and the drilled formation lead to failure of cement, and hence the well integrity can be affected. Furthermore, the prolong interaction between the stored  $CO<sub>2</sub>$  and the casted cement can lead to corrosion of embedded well tubular. This deterioration of the casted well is a major cause of cracking and channeling of the cement matrix resulted in early gas migration. Such problems can lead to early work over jobs or shutting-down some wells that resulted in losing its productivity.

The previous work focused on  $CO<sub>2</sub>$  storage and its effect on enhanced gas recovery. Others studied the interaction of  $CO<sub>2</sub>$  with casting cement and provided solution for its deterioration effect on casted cement. [Le](#page--1-0) [Guen et al. \(2009\)](#page--1-0) and [Pacala and Socolow \(2004\)](#page--1-0) showed that  $CO<sub>2</sub>$  sequestration is one of the most important techniques used to reduce the amount of greenhouse gases in order to protect the environment. [Tarco](#page--1-0)

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[and Asghari \(2010\)](#page--1-0) mentioned that  $CO<sub>2</sub>$  storage can be performed either in abundant or active wells.

The key point in the sequestration operation is the assurance of permanent storage of  $CO<sub>2</sub>$  within the selected formation. Two possible ways of  $CO<sub>2</sub>$  leakage after injection; one is the leakage through the cap rock and the other is the leakage through a low strength bond between cement and permeable formation. Another major challenge associated with  $CO<sub>2</sub>$  sequestration is the injection of low pH brine on casted wells durability [\(Abid et al., 2015\)](#page--1-0). [Agbasimalo and Radonjic \(2012\)](#page--1-0) concluded that Portland cement class H tends to degrade by injecting low pH brine and the degree of degradation increased with the presence of the drilling fluid filter cake. [Li et al. \(2014\)](#page--1-0) concluded that the cement tends to lose its mechanical integrity after long term exposure to sour gases. [Bai et al. \(2016\)](#page--1-0) stated that both mechanical loading and chemical corrosion of cement affect the cement bond with the formation. The special types of cement as the one contains high alumina can be used for high injection rate operation especially with the presence of sour gases. [Condor and Asghari \(2009\)](#page--1-0) have used type 10 classes A and G cement that are recommended for plugging in oil and gas industry. They found that the permeability of cement cubes of both types initially decreased and after a few months increased. This increase in permeability was observed to be fast and high as the temperature increased. The compressive strength reduced for both types after few months. A poor bonding between the cement and the holder was confirmed by measuring the shear and hydraulic bonding. These results confirmed that wellbore leakage is the easy pass for the  $CO<sub>2</sub>$  to escape out of the permeable formation. Moreover, as the cement is exposed to  $CO<sub>2</sub>$ , a carbonation reaction and leaching occurs. These phenomena triggers cement degradation where the rate of leaching is faster than formation of calcium silicate hydrate [\(Lesti et al., 2013](#page--1-0)).





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In order to enhance the casted wells against  $CO<sub>2</sub>$  degradation, [Djwantoro Hardjito and Rangan \(2004\)](#page--1-0) and [Diaz and Allouche \(2010\)](#page--1-0) stated that addition of fly ash and alkaline fluid to class F cement will form a geopolymer cement that has a higher compressive strength and more acid resistance compared with Portland cement. Indeed, geopolymer-based cement was found to be stiffer and has a higher compressive strength than class G cement at higher temperature. [Omosebi et al. \(2015\)](#page--1-0) studied the degradation of Portland cement class G and H under the effect of  $CO<sub>2</sub>$  storage at 38 °C and 42 MPa. Their results showed that the addition of 35 wt% silica to the base cement enhanced the compressive strength for both classes. They concluded that carbonation process enhanced the cement properties while the leaching process compromised the cement integrity. [Daou et al.](#page--1-0) [\(2014\)](#page--1-0) showed that adding self-healing material to  $CO<sub>2</sub>$ -resistant cement will enhance the mechanical properties of cement and improve the cement integrity. The use of self-healing material allows materials to expand and plug any fractures which may be formed due to the reaction of CO<sub>2</sub> with cement.

Although of great efforts that were made to enhance casted well against  $CO<sub>2</sub>$  migration and degradation, little work has been made on studying the interaction of  $CO<sub>2</sub>$  with casted well and especially at a temperature and pressure that simulate the real well conditions. Furthermore, little work has been done on developing a new casting material that has low cost and high  $CO<sub>2</sub>$  resistance and high compressive strength.

Recent review works on geopolymers from clay minerals show that these produced material exhibit stable physical and chemical properties, which can be used as substituent to conventional cements in EOR and EGR ([Liew et al., 2016; Robayo-Salazar et al., 2016](#page--1-0)). Moreover, clay minerals have proved their adsorption affinity for acidic gases such as  $CO<sub>2</sub>$  and H<sub>2</sub>S, which can be used in maintaining  $CO<sub>2</sub>$  in gas and oil wells ([Bkour et al., 2016; Shawabkeh and Harahsheh, 2007;](#page--1-0) [Shawabkeh et al., 2015](#page--1-0)).

This work aims at developing a new material from kaolin that has a high resistance to  $CO<sub>2</sub>$  in upstream for EOR and EGR. The synthesis conditions as well as the adsorption-desorption characteristics at simulated well conditions will be presented. Then a model that simulates the adsorption isotherms and the thermodynamic properties for the interaction between the developed material and the  $CO<sub>2</sub>$  will be investigated.

#### 2. Materials and methods

#### 2.1. Materials

Kaolin clay was collected from Al-Riyadh area in Saudi Arabia. The kaolin rocks were crushed, grinded, and sieved to pass 45 μm and stored in closed containers. Silica sand with purity of 99.9% silica was obtained from Jubail industrial area in Saudi Arabia. The particle size distribution shows 0.045% passed 75 μm, 0.84% (75–150 μm), 5.67% (150–212 μm), 61.84% (212–425 μm), 28.34% (425–710 μm) and the remaining 3.26% was greater than 710 μm. Analytical grade sodium hydroxide obtained from EMD Millipore was freshly prepared with varied concentrations (13–19.7 mol/L). Double distilled water obtained from milli-pore was used for the preparation of solutions.

#### 2.2. Fabrication of treated kaolin material

A series of kaolin treated samples were prepared from solutions of sodium hydroxide, kaolin clay, and silica sand filler as per ANOVA experimental design as shown in Table 1. Homogeneous samples of known amounts of clay minerals and sand were mixed with different ratios of clay, sand, and sodium hydroxide solutions. The synthesis parameters such as mass of clay mineral, sand, NaOH and water were varied according to full statistical analysis design of experiment in order to identify the optimum conditions for the synthesis of the mineral polymeric material ([Kim and Lee, 2013](#page--1-0)). The used amount of water is



Design of experiment for the synthesis of treated kaolin.



limited by the plastic limit of the clay, which is determined to be around 25%. The samples were mixed using Kenwood-type mixer (ModelKM020-China), of 6.7 L capacity, at a speed of 100 rpm for 5 min. Then a higher speed of approximately 200 rpm was allowed for 10 min to assure complete mixing of the constituents. After homogenizing the samples the sodium hydroxide solution is added to the contents of the mixer with continuous mixing.

After completion of mixing with NaOH solution, each sample is divided into 9 (70 g) parts. Specimens were molded immediately in stainless steel cylinders (diameter of 28.3 mm and height of 50 mm) at a pressure of 15 MPa using manually hydraulic press (MEGA 50 ton type press, model KPD 50- Spain). Then, the molded specimens were heated at 80 °C for 24 h in a well-controlled ventilated oven to allow for the reaction between kaolin, silica and sodium hydroxide to take place. After curing, the molded specimens were removed from the oven and allowed to cool at room temperature as shown in Fig. 1.

### 2.3. Pretreatment tests and characterization

The obtained specimens were divided into three groups. Each group contains three specimens. The first group was left for curing at room temperature for seven days; the second group was cured in immersed water for seven days and the third group was used for initial characterization including XRD and TGA analysis.

The X-ray diffraction (XRD) analysis was carried out to identify major crystalline and potentially newly formed phases. The XRD patterns were measured from 5° to 80° 2θ at a scan rate of 2°/min. The crystalline phases were identified by analyzing the positions of the peaks using the software package supplied with the instrument.

Thermogravimetric analysis (TGA) was conducted on STA 409 NETZSCH thermal analyzer to measure the variation of samples mass as a function of temperature in nitrogen environment. Samples of 10 mg were heated at a constant rate of 5°/min and up to 900 °C and the difference in sample mass was recorded.



Fig. 1. Fabricated treated kaolin.

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