Contents lists available at ScienceDirect

Applied Clay Science

journal homepage: www.elsevier.com/locate/clay



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ARTICLE INFO

Article history: Received 24 October 2016 Received in revised form 8 February 2017 Accepted 9 February 2017 Available online xxxx

Keywords: Treated kaolin material Construction Carbon dioxide storage Clay EGR and EOR

1. Introduction

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ABSTRACT

A new material from clay was developed that has a resistance to carbon dioxide flow in upstream for plugging CO_2 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_2 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 CO_2 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_2 sequestrations wells.

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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 CO₂ into atmosphere and recovering the oil and gas that left after secondary processes. However, maintaining CO₂ in gas and oil wells is a challenge in CO₂ 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 CO₂ 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_2 storage and its effect on enhanced gas recovery. Others studied the interaction of CO_2 with casting cement and provided solution for its deterioration effect on casted cement. Le Guen et al. (2009) and Pacala and Socolow (2004) showed that CO_2 sequestration is one of the most important techniques used to reduce the amount of greenhouse gases in order to protect the environment. Tarco

* Corresponding author. E-mail addresses: faqir@ju.edu.jo (N.M. Faqir), elkatatny@kfupm.edu.sa (S. Elkatatny), mmahmoud@kfupm.edu.sa (M. Mahmoud), rshawabk@kfupm.edu.sa (R. Shawabkeh). and Asghari (2010) mentioned that CO₂ storage can be performed either in abundant or active wells.

The key point in the sequestration operation is the assurance of permanent storage of CO₂ within the selected formation. Two possible ways of CO₂ 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 CO₂ sequestration is the injection of low pH brine on casted wells durability (Abid et al., 2015). Agbasimalo and Radonjic (2012) 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) concluded that the cement tends to lose its mechanical integrity after long term exposure to sour gases. Bai et al. (2016) 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) 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 CO₂ to escape out of the permeable formation. Moreover, as the cement is exposed to CO₂, 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).





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In order to enhance the casted wells against CO₂ degradation, Diwantoro Hardiito and Rangan (2004) and Diaz and Allouche (2010) 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) studied the degradation of Portland cement class G and H under the effect of CO₂ 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. (2014) showed that adding self-healing material to CO₂-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_2 with cement.

Although of great efforts that were made to enhance casted well against CO_2 migration and degradation, little work has been made on studying the interaction of CO_2 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_2 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). Moreover, clay minerals have proved their adsorption affinity for acidic gases such as CO_2 and H_2S , which can be used in maintaining CO_2 in gas and oil wells (Bkour et al., 2016; Shawabkeh and Harahsheh, 2007; Shawabkeh et al., 2015).

This work aims at developing a new material from kaolin that has a high resistance to CO_2 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_2 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 μ m 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 μ m, 0.84% (75–150 μ m), 5.67% (150–212 μ m), 61.84% (212–425 μ m), 28.34% (425–710 μ m) and the remaining 3.26% was greater than 710 μ m. 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). The used amount of water is

Table 1

Design of experiment for the synthesis of treated kaolin.

Factor	Component	Level	Lower & upper values (g)
А	Clay	A1	100
		A2	125
В	Sand	B1	125
		B2	150
С	NaOH	C1	13
		C2	17
D	Water	D1	19
		D2	25

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° 2 θ 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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