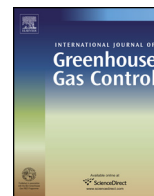




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A new polymer nanocomposite repair material for restoring wellbore seal integrity

Moneeb Genedy^a, Usama F. Kandil^b, Edward N. Matteo^c, John Stormont^a,
Mahmoud M. Reda Taha^{a,*}

^a Department of Civil Engineering, University of New Mexico, MSC01 1070, 1 University of New Mexico, NM 87131-0001, USA

^b Polymer Nanocomposite Center of Excellence, Egyptian Petroleum Research Institute (EPRI), Cairo 11727, Egypt.

^c Sandia National Laboratories, Albuquerque, NM, USA

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ABSTRACT

Seal integrity of functional oil wells and abandoned wellbores used for CO₂ subsequent storage has become of significant interest with the oil and gas leaks worldwide. This is attributed to the fact that wellbores intersecting geographical formations contain potential leakage pathways. One of the critical leakage pathways is the cement-shale interface. In this paper, we examine the efficiency of a new polymer nanocomposite repair material that can be injected for sealing micro annulus in wellbores. The bond strength and microstructure of the interface of Type G oil well cement (reference), microfine cement, Novolac epoxy incorporating Neat, 0.25%, 0.5%, and 1.0% Aluminum Nanoparticles (ANPs) with shale is investigated. Interfacial bond strength testing shows that injected microfine cement repair has considerably low bond strength, while ANPs-epoxy nanocomposites have a bond strength that is an order of magnitude higher than cement. Microscopic investigations of the interface show that micro annulus interfacial cracks with widths up to 40 μm were observed at the cement-shale interface while these cracks were absent at the cement-epoxy-shale interface. Fourier Transform Infrared and Dynamic mechanical analysis measurements showed that ANPs improve interfacial bond by limiting epoxy crosslinking, and therefore allowing epoxy to form robust bonds with cement and shale.

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1. Introduction

Seal integrity of both production wells and abandoned wells is critical to a variety of subsurface applications, including geologic storage of CO₂. Loss of zonal isolation is a serious issue and is integral to ensuring protection of human health and the environment. Wellbores intersecting geological formations contain potential leakage pathways that exist due to either well-completion process or degradation of the cement and steel casing (Zhang and Bachu, 2011). Among the potential leakage pathways in wellbores as summarized schematically in Fig. 1 (Celia et al., 2005), the steel-cement and rock-cement interfaces are two of the most critical leakage pathways (Carey et al., 2007). While considerable attention has been given to the steel-cement interface (Baldan, 2004; Nakayama and Beaudoin, 1987; Zhai et al., 2006), little effort has been focused on studying the rock-cement interface. This article focuses on repair of the rock-, specifically shale, cement interface for seal integrity. Due to their low permeability, shale layers often

serve as cap rocks for underlying hydrocarbon reservoirs. Ensuring the integrity of wellbores at locations where they penetrate these layers is important for the containment and control of reservoir fluids.

Prior investigations have shown that the cement-shale bond is affected by both cement and shale. Cement shrinkage and growth of the calcium hydroxide crystals at the interfacial zone of cement-bonded surfaces are well-known reasons to weaken cement bond with most interfaces including the cement-shale interface (Nakayama and Beaudoin, 1987; Mehta and Monteiro, 2013). Moreover, the swelling coefficient of the shale formation has a significant impact on interfacial bond between the cement and shale. The higher the swelling coefficient, the weaker the bond-strength is between the cement and shale (Ladva et al., 2005). The morphology of the rock surface also significantly affects the bond strength of the cement-shale interface. Shale formation with higher surface roughness would tend to have higher bond strength with cement compared with smooth shale formations (Nasir and Fall, 2008). The presence of carbonated brine can significantly deteriorate cement and affect the wellbore seal integrity (Carey et al., 2007; Duguid and Scherer, 2010; Huet et al., 2010; Kutchko et al., 2007, 2008; Liteanu et al., 2009; Matteo and Scherer, 2012; Rimmelé

* Corresponding author.

E-mail address: mrtaha@unm.edu (M.M. Reda Taha).

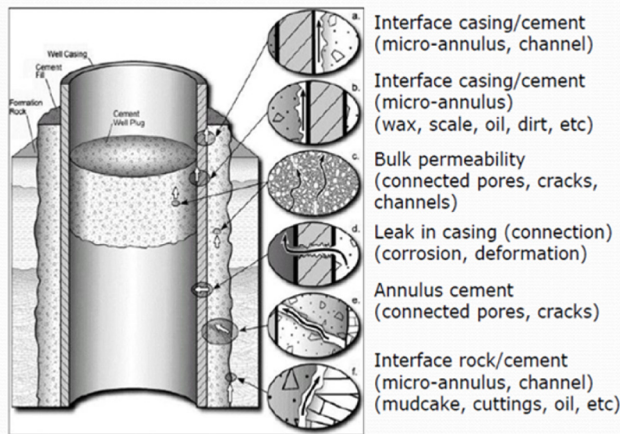


Fig. 1. Schematic representation of zonal isolation and potential leakage pathways (Celia et al., 2005).

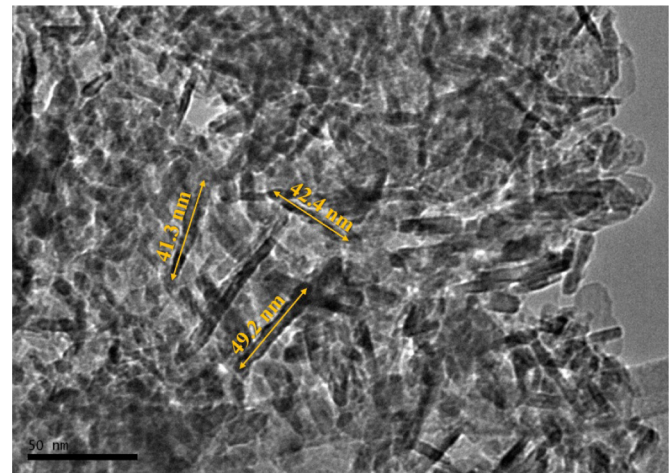


Fig. 2. TEM micrograph showing aluminum nanoparticles and their size.

et al., 2008). The above factors favor debonding between cement and shale over the long-term.

Repair of the cement-shale bond requires injecting a repair material that can fill the interfacial contact and improve seal integrity. A repair material should have a relatively low viscosity so it can flow during injection to penetrate microannulus cracks (micro cracks along a cement-shale contact interface). Such material should have minimal shrinkage to reduce the likelihood that a new flow path will develop between the repair material and the cement and/or shale. Furthermore, the repair material should also be flexible to prevent debonding due to further cement shrinkage, shale swelling, temperature changes and/or shock waves.

We suggest that polymer based materials are optimal repair materials to achieve these requirements. Polymers have shown very high bond strength with metallic surfaces (Baldan, 2004). Thermoset polymers, specifically Novolac-based epoxies, show very high bond strength with different surfaces. Moreover, incorporating nanomaterials such as Aluminum Nanoparticles (ANPs) in polymers was able to improve their mechanical properties and durability (Pocius, 2012; Salemi and Behfarnia, 2013; Shokrieh et al., 2012; Wetzal et al., 2006). Zhai et al. (2006) reported improved bond between polymer matrices and metallic surfaces when nanomaterials were dispersed in the polymer matrix. Our prior investigations showed that Novolac epoxy incorporating ANPs significantly improved the bond strength of the cement-steel interface (Genedy et al., 2014).

In this study, we investigate the efficiency of using Novolac epoxy incorporating ANPs as a repair material for sealing cement-shale microannulus. The bond strength of ANPs-epoxy nanocomposites incorporating different content of ANPs is investigated using push-out tests. In these tests, ANPs-epoxy nanocomposites were injected into an 800 μm artificial gap created between an inner shale cylinder and an outer cured cement socket. The ANPs-epoxy nanocomposites were compared to a standard microfine cement repair material, typically used in the industry, which was injected to fill the same above gap. The bond strengths of ANPs-epoxy polymer nanocomposites as well as the microfine cement repair materials were compared with the bond strength of a reference case where Type-G oil well cement (OWC) was cast around shale and fully cured to achieve the highest possible bond strength. Microstructural analysis including Dynamic Mechanical Analysis (DMA) and Fourier Transform Infrared (FTIR) analysis as well as light microscope investigations were conducted to understand the significance of nanomaterials on epoxy behavior.

2. Experimental methods

2.1. Materials

The shale used in this investigation was Mancos Shale. Type G (API Class G) OWC was used as the reference cement material. Microfine cement, generally used to repair cracks in oil wells, as provided by the manufacturer was used. The microfine cement has a composition that is comparable to high sulfate-resistant cements and grain size (d_{95}) of 9.5 μm . The mix used for the microfine cement has water to cement ratio (w/c) of 0.7 and 2.0% of the dry cement weight super plasticizer (recommended by the manufacturer). Novolac epoxy was selected as the repair material due to its high chemical resistance, relatively high thermal performance and good flowability. Novolac epoxy system is a low viscosity cycloaliphatic polyamine blend with low viscosity hardener. Researchers showed water, acids (such as carbonated water) and saline solutions to have negligible effect on hardened Novolac epoxy (Atta et al., 2008; Tchoquessi Doidjo et al., 2013; Soares-Pozzi and Dibbern-Brunelli, 2016). ANPs were used to modify the epoxy matrix. ANPs are Aluminum Oxide Al_2O_3 with maximum particle size of 50 nm were obtained from Sigma Aldrich, Inc. Microscopic image of ANPs using transmission electron microscope (TEM) is shown in Fig. 2. Three weight contents of ANPs were examined in addition to neat epoxy being 0.25%, 0.50% and 1.0% by weight of the epoxy resin.

2.2. Polymer nanocomposite preparation

To prepare the polymer nanocomposite, the ANPs were added to the required amount of the resin, and the mix was stirred for 2 h at 110 $^\circ\text{C}$ using magnetic stirring. This relatively high mixing temperature was used to reduce the resin viscosity and improve the dispersion of ANPs. The mix was then sonicated for 2 additional hours at 65 $^\circ\text{C}$ using an ultrasonic homogenizer. The polymer resin nanocomposite was left to cool and reach room temperature and then mixed with the hardener with mixing ratio 2.2:1 (Resin:Hardener) for 3 min using low speed mixer until a uniform mix was obtained. It is important that the nanomaterials are very well dispersed in the mixture to prevent gravity settling. This was achieved by the interaction of ANPs with the polar groups of the epoxy resin.

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