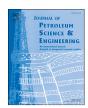
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Novel methodology to evaluate displacement efficiency of drilling mud using fluorescence in primary cementing



Myoungsung Choi^{a,*}, George W. Scherer^b, Robert K. Prudhomme^c

- ^a Dept. of Safety Engineering, Dongguk University-Gyeongju, 123 Dongdae-ro, Gyeongju, Gyeongbuk, South Korea
- b Dept. of Civil and Environmental Engineering, Princeton University, Princeton, NJ, 08544, USA
- ^c Dept. of Chemical and Biological Engineering, Princeton University, Princeton, NJ, 08544, USA

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ABSTRACT

For primary cementing of a well, successful displacement of drilling mud from the casing and annulus, and properly conditioning those surfaces to bond with the cement slurry, are paramount to achieve zonal isolation. In this study, to evaluate displacement of drilling mud from well casings, a fluorescence methodology incorporating a hydrophobic dye was developed. Fluorescence is attractive because the dye is highly oil soluble and non-polar and can be detected at very low concentrations, so chemical interference between dye and drilling mud is minimized. From the fluorescence measurements, the thickness of residual drilling mud can be quantitatively determined, which makes it possible to quantify the efficiency of drilling mud removal. Residual oil layers under consideration in this study from $32~\mu m$ to $1.5~\mu m$ are observed. The spacer fluids D and E are excellent in their ability to remove the drilling mud, whereas spacer F performs poorly. The effects of metallurgy and surface roughness on wettability were investigated using the measured thickness of residual drilling mud. There is little effect of the composition of the steel tube on wettability, but the surface roughness or presence of corrosion can significantly affect the wettability.

1. Introduction

Successful displacement and effective removal of oil-based or synthetic-based drilling mud from the wellbore prior to cementing is critical to develop an excellent bond between casing and formation, which leads to successful zonal isolation. The drilling mud can leave a thin layer of oil on the casing and the formation that can prevent the cement slurry from forming a strong bond with the formation and the casing, preventing complete structural integrity. Thus, the evaluation of successful displacement of drilling mud from the casing and annulus, and properly conditioning those surfaces to bond with the cement slurry, are paramount.

The casing and formation should be water-wet for proper cement bonding to occur. For that purpose, properly designed displacement fluids incorporating surfactants (spacer systems) are normally applied between drilling mud and cement slurry to effectively displace the oil layer and allow maximum recovery from the oil-wet surface condition. These displacement fluids thereby allow effective annular sealing with the typical cement slurries. The present study addresses the evaluation of displacement of drilling mud in terms of wettability.

The terminology of wettability is extensively used to evaluate the surface condition (Schrader and Loeb, 1992; De Gennes, 1985; Johnson, 1993; Good, 1992; Quere et al., 2008; Dezellus and Eustathopoulos, 2010; Rowlinson and Widom, 1982). Wettability describes the preference of a solid to be in contact with one fluid rather than another, which aptly describes the balance of surface and interfacial forces. Wettability studies usually involve the measurement of contact angle as the primary data, which indicates the degree of wetting when a solid and liquid interact (Zisman and Fowkes, 1964; Chen et al., 2005; Tadmor, 2004; Förch et al., 2009; Yuan et al., 2013). However, to evaluate the displacement, there have been few studies to directly determine the residual oil layer thickness in primary cementing. In addition, for heterogeneous rough materials, such as the casing in an oil well, the wettability assessment is made more difficult by local variations (Schneider et al., 2011).

Therefore, in the present study, a novel methodology using a hydrophobic fluorescence dye dissolved in drilling mud was developed to directly evaluate the residual oil layer thickness on the surface of the casing after primary cementing. Moreover, to examine the effects on wettability of the various types of casing used in actual wellbores, the

E-mail address: mschoi@dongguk.ac.kr (M. Choi).

^{*} Corresponding author.

effect of metallurgy and surface roughness of metal tubes were quantitatively investigated.

2. Principle of the fluorescence methodology

To quantify the amount of drilling mud left on the surface of the metal tube following exposure to the spacer, a hydrophobic fluorescence dye is mixed into the drilling mud; after a pipe is exposed to the dyed drilling mud followed by spacer, the residual drilling mud on the metal tube is flushed out with a solvent, and the fluorescence of that solution is measured by fluorescence spectroscopy (Rendell, 1987; Sharma and Schulman, 1999; Gauglitz and Vo-Dinh, 2003; Lakowicz, 1999).

In fluorescence spectroscopy, the dye is excited into a higher energy state by absorbing a photon. Collisions with other molecules cause the excited molecule to lose vibrational energy and drop into a lower energy state. This process is visualized with the Jablonski diagram in Fig. 1 (Elumalai et al., 2002; Jabloński, 1933). As a molecule drops into a lower energy level, it emits a photon (Jabloński, 1933). As molecules may drop down into any of several energy levels, the emitted photons will have different energies, and thus different frequencies. Fluorescence spectroscopy measures the relative intensities of light emitted at each frequency.

Fluorescence is attractive, because the presence of a dye can be detected at ppb (part per billion) levels (Lakowicz, 1999; Rendell, 1987; Sharma et al., 1999). This sensitivity enables the addition of very small amounts of dye, so chemical interference between the dye and the sample under consideration can be minimized.

The quantity of interest is the amount of oil that remains on the metal surface after displacement by the spacer. A fluorescent dye is chosen that is highly oil soluble, and highly water insoluble: Nile red. It is non-polar, so it is not likely to strongly interact with other additives in the oil-based drilling muds. After the displacement experiment, the dye is solubilized with tetrahydrofuran (THF), a water-miscible organic solvent.

3. Experimental programs

3.1. Materials and sample preparation

The drilling mud used was a synthetic oil-based system (BAKER HUGHES, Rheo-logic) having a density of $1105\,\mathrm{kg/m^3}$. To evaluate the displacement of the oil layer from the metal tube, three types of spacers designed with a density $1740\,\mathrm{kg/m^3}$ were compared and those proportions are in Table 1. In the process of primary cementing, to examine the effects of the cement slurry stage on removal of the oil layer, cement slurries were prepared according to the API RP 10B-2 (which includes methodology to verify the adequacy of the cementing fluids for the well

Table 1Proportions of spacer systems (Unit: kg/m³).

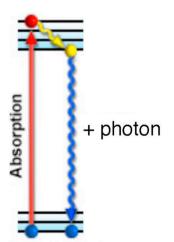
Compositions	Spacer D	Spacer E	Spacer F
Barite	948	1123	968
Deionized Water	725	_	730
Solvent	-	617°	-
Spacer Blend D	31 ^a	-	-
Spacer Blend F	_	_	4^{d}
Surfactant	37 ^b	-	38 ^e

- ^a Polysaccharides and Water-swellable Clays.
- ^b Ethoxolated Nonophenols.
- ^c Mixture of Petroleum Distillates, Alcohols, and Benzenesulfonic Acid.
- ^d Polysaccharides and Water-swellable Clays.
- e Ethoxylated Alcohols.

cementing industry), using distilled water and a class H oil well cement. The density of the cement slurry was 1968 kg/m 3 and the ratio of water to cement was 0.37. The compositions of the cement slurries and the concentration of additives are shown in Table 2. Solid additives were blended with the cement powder. Liquid additives were added to the water prior to mixing with solid additives. To investigate the effect of metallurgy on wettability, two different types of steel tubes that are widely used in oil wells, N80 and P110, having diameters of 1.25 and 2 inches, respectively, were prepared. The metal tubes were prepared with two types of surface condition, clean and corroded, to examine the effects of surface roughness on wettability.

Table 2
Compositions of cement slurry.

Compositions	Weight up (kg/ m³)	Concentration (%)	Chemical Base	Note
Cement	1403		_	Class H
Water 52	522	_	_	Deionized
				Water
Antifoam 2.5	2.5	0.02	Silicone	Liquid
		Derivatives	Additive	
Dispersant 4.3	4.3	0.03	Naphthalene	Liquid
			Sulfonate-	Additive
			formaldehyde Co	
			polymer	
Fluid Loss 11.7	11.7	0.1	Synthetic	Liquid
			Copolymer	Additive
Retarder 5.7	5.7	0.04	Lignosulfonate	Liquid
				Additive
Potassium	15.7	0.03	-	Solid
Chloride				Additive





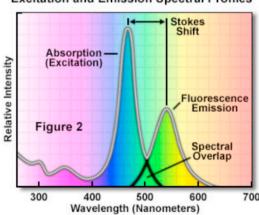


Fig. 1. Diagram for fluorescence principle [19].

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