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Properties of oil well cement with high dosage of metakaolin

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HIGHLIGHTS

- At 75 °C, the early-term strength of cement decreased with the increasing MK content.
- At 75 °C, the set cement with more MK showed higher long-term strength.
- Beyond 150 °C, samples M40 to M60 presented good strength in all test ages.
- Hydration products and structure would influence strength and porosity of cement.
- High dosage of MK cement is suitable for corrosion and high temperature conditions.

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ABSTRACT

This research work was carried out to evaluate the properties of oil well cement with high dosage of metakaolin. The fresh pastes were made using an initial water/solid ratio of 0.5 by weight and then hydrated for periods up to 56 days. The mechanical performance was assessed from compressive strength. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were applied in order to study the hydration products and qualitative of microstructure. Feasibility as oil well cement of metakaolin replaced cement was assessed by thickening time, corrosive fluid exposure, density and rheology test. Test results confirmed that the early-term strength decreased with the increasing metakaolin content and the set cement with more metakaolin showed higher long-term strength at 75 °C. The cement pastes with 40–60%wt metakaolin presented good strength both in early-term and long-term curing at 150 °C and above. The metakaolin replaced cement pastes demonstrated low porosity and good strength due to stable hydration products and dense microstructure. Meanwhile, the filling effect of metakaolin partials reduced porosity and improved corrosion resistance of hardened pastes. The MK replaced cement pastes are applicative for the cementation of deep wells, acid rich wells and geothermal wells.
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1. Introduction

Portland cement has been used as well cement for many years. However, Portland cement is not suitable for some critical circumstances, especially in deep oil wells, geothermal wells, and acid rich wells. It goes through strength reduction, increased porosity, and there are durability concerns in high temperature and corrosion environments where Portland well cement could lose its sealing property in a short period of time [1–4]. Elevated temperatures lead to gradual damage of connections between admixture and cement paste while causing the generation of mineral with poor structure, resulting in bad mechanical behavior [5]. Corrosive fluids migrate in cracks and structure pores in cement sheath, which lead to the increase of permeability and porosity. As a result, an increase of area and contact time between the cement and corrosive fluids occurs, accelerating the corrosion procedure [6].

Presently, pozzolanic material had already proved to be a material which leads to high strength in early age, low porosity, high temperature resistance and good chemical corrosion resistance [7–9]. These materials react with $Ca(OH)_2$ which is released in the hydration of cement and convert $Ca(OH)_2$ into high crystallinity CSH (calcium silicate hydrates) phases according to pozzolanic reaction. The most common pozzolanic materials are fly ash (FA), silica fume (SF) and metakaolin (MK). Due to the advantageous capabilities, the pozzolanic material is a prospective substitution to ordinary Portland well cement. However, the stability of performance and market price should be considered in cementing operation. The properties of FA are distinct in different batches

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Table 1
Chemical composition and physical properties of OPC.

Oxides	Wt%
CaO	64.20
SiO_2	19.40
Fe_2O_3	5.50
Al_2O_3	4.50
SO_3	2.80
MgO	2.00
Na_2O	0.10
K_2O	0.60
Loss on ignition	0.49
Density (g/cm^3)	3.15
Specific surface area (m^2/kg)	336.00

Table 2
Chemical composition and physical properties of MK.

Oxides	Wt%
CaO	0.17
SiO_2	55.06
Fe_2O_3	0.76
Al_2O_3	42.12
SO_3	0.15
MgO	0.06
Na_2O	0.06
K_2O	0.55
Loss on ignition	1.20

because FA is manufactured from coal combustion [10,11]. Meanwhile, the price of SF which is more than 290$ per ton is not economical for large quantity cementing operation. Unlike other pozzolans, MK, obtained by thermal treatment of kaolin, is much more inexpensive than SF. Its price is approximately 140$ per ton and the performance of MK is stable. In addition, the substitution of MK also can reduce the pollution of cement industry [12].

Currently researches usually use MK as a cement substitute with 5–20%wt [13–16]. Shatat et al. [13] incorporated the ternary blends of cement with 5–10%wt rice husk ash and 15–20%wt MK. The hardened cement blend showed better mechanical behavior than ordinary Portland cement. Morsy et al. [14] studied the behavior of cement containing nano-MK at elevated temperatures. They indicated that replacement of cement with 5%wt nano-MK at 25 °C leads to optimal mortar and 15%wt nano-MK replacement at 250 °C–800 °C gives better strength. Li et al. [17] compared MK cement with alkali-activated slag cement, they indicated that cement pastes containing MK get better strength at high temperatures.

However, the performance in well cementation of oil well cement pastes containing high dosage of MK (more than 20%wt) is rarely reported. Using greater proportion of MK could significantly reduce the environmental impact of cement industry. Meanwhile, if the properties of high MK dosage replaced cement are equal or even better than low MK dosage cement, the high dosage of MK replaced cement will be a promising candidate as an alternative to oil well cement. Brooks et al. [18] reported that setting time significant decrease as the dosage of MK increase. They explained this phenomenon is caused by the increasing water demand. This may be the bottleneck reason for not using high dosage of MK in the industry. In a recent research by the authors, the setting time issue with high MK dosage was solved by adding specific retarder and adjusting water-solid ratio. So it is possible to use high dosage of MK replaced cement in the industry. Therefore, this study aimed to produce cement mixture containing high dosage of MK which is suitable for deep well, geothermal well and acid rich well cementation. The properties of Portland cement pastes containing high dosage of MK were studied. This was done according to determination of compressive strength, porosity, hydration products and microstructure test. Moreover, the corrosion-resistance, thickening time, rheological property and density of MK replaced cement pastes were measured.

2. Materials and methods

2.1. Materials

The following raw materials were used: API class G oil well Portland cement (OPC) was obtained from Shandong Shengwei Enterprise Company (Linqu, China). The chemical composition and physical properties of OPC are presented in Table 1. This cement was used in preparing all samples. MK was obtained from Jiaozuo Yukun Enterprise Company (Jiaozuo, China). Chemical composition and physical properties of MK are shown in Table 2. Fluid loss control additive BXF-200L was obtained from Company of CNPC (Tianjin, China). This additive reduces the fluid loss. Dispersing agent FHJZ-1 obtained from Fuhai Industry Development Company (Dongying, China), was used to reduce the friction between particles. Retarder OAI, synthesized by Shenglai Guo, was used to adjust the thickening time of cement slurry. The primary functional groups of OAI are maleic acid and itaconic acid.

2.2. Methods

2.2.1. Manufacturing process

Cement slurries were mixed according to API Standard 10B-3-2004. Additives replaced the cement in a certain proportion by weight. Mix design parameters of pastes and their designation are given in Table 3.

First, the fluid loss control additive, retarder and dispersing agent were dissolved in water and transferred into the cup of waring blender. Then, the blended cement was added within 15 s to the aqueous solution with a stirring rate of 4000 rpm and mixed for 35 s at 12,000 rpm. After being prepared, cement slurry was placed into 5 cm cubes for compressive strength test and pressurized consistometer for consistency measurement.

2.2.2. Compressive strength test

Considering the different in downhole conductions, temperature would be an important factor that affects cementing quality. Cement strength decreases when the temperature reaches 110 °C. In some cases, the temperature could even reach 240 °C. For example, a reservoir in Villafortuna Trecate oilfield has been identified between 5800 and 6100 m depth with a temperature beyond 150 °C [19]. Another reservoir at depths of 5641–6027 m and temperature beyond 230 °C in Well ND1 in the Jizhong oilfield has been discovered [20]. So different temperature intervals were set and the molds were placed into a high temperature chamber. Cement slurries were cured for different time periods at 75 °C, 150 °C, 240 °C, respectively. The

Table 3
Mix design parameters and their designation.

Mixes	Mix proportion (Wt%)		Water/solid ratio	Fluid loss additive (Wt%)	Dispersing agent (Wt%)
	OPC	MK			
M0	100	0	0.5	4	0.00
M30	70	30	0.5	4	1.50
M35	65	35	0.5	4	1.75
M40	60	40	0.5	4	2.00
M45	55	45	0.5	4	2.25
M50	50	50	0.5	4	2.50
M55	45	55	0.5	4	2.75
M60	40	60	0.5	4	3.00

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