

## Laboratory Study on Optimization of Oil-Soluble Cement Material

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### Abstract

A new cement slurry system with strong selective permeability to oil and water, namely oil-soluble cement system, was introduced. It is composed of three kinds of basic agents including pore-enhancing grains, pore-connecting agent and phase-permeability enhancing agent. The pore enhancing grains-ZKJ-1, which can broaden the pore significantly was obtained from many natural oil soluble organics. In order to further interconnect the cement stones' pores and enhance the selective permeability to oil and water, the YRJ was selected as the connecting agent and phase-permeability enhancing agent. Combined with three other agents, the cement slurry has a good selective permeability to water and oil and high strength.

**Key words:** Oil solubility; Cement additive; Effective permeability; Laboratory testing

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### INTRODUCTION

With the further development on low permeability reservoir and oil and water interlayer, large pressure difference resulted by Conventional cementing will create severe pollution on low permeability reservoir and fractured hydrocarbon reservoir, which will also reduce well production in above mentioned reservoir<sup>[1,2]</sup>.

In addition, long-term water injection leads high water cut in hydrocarbon reservoir, so that some oil wells were shut down<sup>[3, 4]</sup>. Oil-soluble cement stone has become a choice, the oil soluble material inside this cement stone can dissolve spontaneously under the action of reservoir oil, and seepage flow path for oil would be formed. However, it cannot dissolve in formation water. Therefore, the research and development of oil-soluble cement system could have many practical applications on low permeability and fractured reservoir cementation, profile control and water plugging, sand control and hydrocarbon reservoir protection. In this paper, we use two main parameters, oil phase permeability and compressive strength of cement stone, to evaluate the new cement system. Three kinds of basic agent including pore-enhancing grains, pore-connecting agent and phase-permeability enhancing agent were optimized by experimental way.

### 1. COMPOSITION OF OIL-SOLUBLE CEMENT SYSTEM

Oil-soluble cement system developed from permeable cement used in sand control, has a strong selective permeability to oil and water. It could form oil seepage flow path spontaneously in formation oil under the action of pore-enhancing grains, pore-connecting agent and phase-permeability enhancing agent.

Pore-enhancing grains could improve the macro pore structure to increase the porosity of cement stone. Pore-connecting agent could increase the cement stone permeability by improving the capillary channel and connecting the macro pore formed by pore-enhancing grains<sup>[5, 6]</sup>. Phase-permeability enhancing agent, which could reduce percolation resistance to oil and increase percolation resistance to water by changing the surface of macro pore and capillary channel from water wet into oil wet, is to make the cement stone have a selective permeability to oil and water.

## 2. EXPERIMENT PRINCIPLES AND METHODS

### 2.1 Experiment Principles

Based on literature researches, we selected some kinds of material with good performance as pore-enhancing grains, pore-connecting agent and phase-permeability enhancing agent to perform laboratory studies. According to the requirements of cementing engineering, the oil phase permeability and compressive strength of cement stone were used as primary indicators to optimize the three basic agents mentioned above.

### 2.2 Material and Reagent

G class cement powder, kerosene, absolute alcohol, YRJ, hollow fiber, viscose, silica sand, aluminum powder, HPAM, SP-80, ZKJ-1, ZKJ-2, ZKJ-3 and SWJZ-1

### 2.3 Experiment Methods

#### 2.3.1 Measurement of Solubility

Pore-enhancing grains were added and mixed with solution under a certain time and temperature. Solubility could be calculated by filtration, drying and weighting. The formula of solubility is:

Solubility = (1- residual mass after dissolution/mass before dissolution)100%

#### 2.3.2 Test Method for Permeability

The cylindrical shaped specimens of cement were removed from molds. Two end faces of specimens should be machined smooth and flat. Then these specimens were transferred to core analysis lab to measure water and oil permeability. The specimens were evaluated after curing at 60 °C, 70 °C, 80 °C and 90 °C for 48 hours. Brine with TDS of 3910 mg/L and density of 1.001 g/cm<sup>3</sup> (at 25 °C) was used to measure water phase permeability, kerosene was used to measure oil phase permeability.

### 2.3.3 Test Method for Compressive Strength After Oil or Water Displacement

The compressive strength after oil or water displacement is the compressive strength of cement stone core measured after oil or water permeability test. The set cement dried in drying oven after permeability test should be processed into test specimens (diameter: 2.50 ± 0.10 cm; length: 2.50 ± 0.10 cm). Both end faces should be ground flat and kept parallel. Then the specimens are placed in a hydraulic press where increasing force is exerted on each specimen until failure. The compressive strength of specimens is obtained through this failure experiments.

## 3. EXPERIMENTAL RESEARCH ON MATERIAL OPTIMIZATION

### 3.1 Optimization for Pore-Enhancing Grains

Three kinds of oil-soluble resin (ZKJ-1, ZKJ-2 and ZKJ-3) were selected as pore-enhancing grains initially after a large number of literature researches and tentative experiments.

#### 3.1.1 Solubility Test for Pore-Enhancing Grains

Solubility is one of the most important parameters for evaluation of pore-enhancing grains. Pore-enhancing grains with good performance should have a high oil solubility and strong hydrophobic nature. It could dissolve in crude oil existing in underground formation easily and has a very low solubility in formation water because of its strong hydrophobic nature. As Table 1 showing, three kinds of resins exhibit a high solubility in kerosene and don't dissolve in distilled water and cement slurry filtrate. That means these resins possess the property of oil-soluble and water-insoluble and could meet requirements mentioned above.

**Table 1**  
**Solubility of Pore-Enhancing Grains in Different Solutions**

Items	Solubility, %		
	ZKJ-1	ZKJ-2	ZKJ-3
Soaked in kerosene for 0.5 hours @ 75 °C	87.61	80.40	80.33
Soaked in kerosene for 1.0 hour @ 75 °C	88.96	82.86	81.40
Soaked in kerosene for 4.0 hours @ 75 °C	89.04	85.04	82.50
Soaked in kerosene for 8.0 hours @ 75 °C	89.93	87.18	83.13
Soaked in distilled water for 10.0 hours @ 75 °C	Insoluble	Insoluble	Insoluble
Soaked in cement slurry filtrate for 10.0 hours @ 75 °C	Insoluble	Insoluble	Insoluble

#### 3.1.2 Influence of Pore-Enhancing Grains on Cement Stone Properties

The oil phase permeability and compressive strength of cement stone after oil displacement are two important parameters for Pore-enhancing grains selection. Excellent

pore-enhancing grains could increase the oil phase permeability obviously and have smaller damage to the compressive strength of cement stone. The influence of ZKJ-1, ZKJ-2 and ZKJ-3 on cement stone properties is shown in Table 2.

**Table 2**  
**Influence of Pore-Enhancing Grains on Cement Stone Properties**

Pore-enhancing grains	Oil phase permeability, $10^{-3} \mu\text{m}^2$				Compressive strength after oil displacement @ 90 °C, MPa
	60 °C	70 °C	80 °C	90 °C	
ZKJ-1	0.23	0.64	1.58	3.65	9.78
ZKJ-2	Very low	Very low	0.22	0.46	10.10
ZKJ-3	Very low	Very low	0.18	0.38	10.86
No Pore-enhancing Grains	No kerosene was driven out from cement stone core after 72 hours displacement				
Formula	Water-cement Ratio 0.6, Pore-enhancing Grains 30%, Silica Sand 40%, Polypropylene Fiber 0.3%, SWJZ-1 0.5%				

Note: "very low" means the permeability is too low to measure accurately.

Table 2 shows that all the resins can increase oil phase permeability of cement stone in varying degrees under a certain temperature range and the oil phase permeability increases as temperature rises. The cement stone with ZKJ-1 has the biggest oil phase permeability. Its compressive strength is slightly smaller than that of cement stones with ZKJ-2 and ZKJ-3. So ZKJ-1 was selected as the pore-enhancing grains after comprehensive balance.

### 3.2 Optimization for Pore-Connecting Agent

#### 3.2.1 Material Selection of Pore-Connecting Agent

Pore connecting material used in permeable cement system is divided into 3 groups including air-entraining

agent, liquid organic and fiber material. The author chose 5 types of materials as alternative material of pore-connecting agent. Air-entraining agent is aluminum powder; liquid organics are absolute alcohol and YRJ<sup>[7, 8]</sup>; fiber materials are hollow fiber and viscose. Considering cement stone permeability could be changed by optimization of grain composition, we also chose silica sand with 0.3-0.5 mm diameter as pore-connecting agent.

#### 3.2.2 Influence of Pore-Connecting Agent on Cement Stone Properties

Table 3 shows the results of influence of pore-connecting agent on cement stone properties.

**Table 3**  
**Influence of Different Pore-Connecting Agents on Cement Stone Properties**

Pore-connecting agent	Dosage of pore-connecting agent, %	Oil phase permeability of cement stone, $10^{-3} \mu\text{m}^2$				Compressive strength after oil displacement @ 90 °C, MPa
		60 °C	70 °C	80 °C	90 °C	
No pore- connecting Agent	0	0.10	0.16	0.55	1.06	8.26
Hollow Fiber	0.3	0.17	0.57	0.72	1.27	8.43
Viscose	0.3	0.50	0.84	1.19	1.51	8.36
Absolute Alcohol	2.0	N/D	N/D	N/D	N/D	N/D
Aluminum Powder	0.7	77.79	227.38	502.62	524.56	2.46
YRJ	5.0	9.28	13.08	15.84	17.51	7.32
Silica Sand	40.0	0.25	0.59	1.10	2.35	8.58

Note. 1) "N/D" means measurements were not done; 2) Dosage of pore-connecting agent is the mass percent of pore-connecting agent to cement powder.

From Table 3 we can see:

(a) Aluminum powder (air-entraining agent) has the best connecting effect. YRJ takes second place. Silica sand, hollow fiber and viscose have poor connecting effect. Cement slurry mixed with absolute alcohol didn't become hard in two days curing time. That means the absolute alcohol has a strong impact on cement hydration.

(b) Cement stone cores mixed with aluminum powder have the lowest value of compressive strength after kerosene displacement. That means aluminum powder could cause serious damage to the compressive strength of cement stone, therefore it is not selected as pore-connecting agent.

(c) Cement stone containing YRJ exhibits a small reduction on compressive strength after displacement than cement stone without pore-connecting agent.

(d) The compressive strength of cement stone mixed with fiber or silica sand is slightly higher than that of cement stone without pore-connecting agent.

After comprehensive consideration of oil-phase permeability and compressive strength after oil displacement, YRJ was chosen as the pore-connecting agent of oil-soluble cement system.

### 3.3 Optimization of Phase-Permeability Enhancing Agent

#### 3.3.1 Material Selection of Phase-Permeability Enhancing Agent

The research on phase-permeability enhancing agent is still in initial discovery phase at present. The function mechanism of this kind of agent is not clear. Based on literature research, there are three kinds of materials that could change the phase permeability of cement stone. The materials are emulsifier, water plugging agent and alcohol. The influence mechanism of these three materials on permeability is elaborated as follows.

##### (a) Emulsifier<sup>[9]</sup>

When the hydrocarbon molecules of emulsifier

interact with free water and bound water in cement stone at the condition of high temperature and high alkaline, emulsification would occur. On the one hand, emulsified liquid could interconnect pores during cement hydration. On the other hand, emulsified liquid could form liquid film with high viscous force and high strength, which could increase flow resistance. Liquid inside the pores of cement stone cannot move until it overcomes flow resistance.

(b) Water Plugging Agent<sup>[10]</sup>

HPAM is always used to plug water in oil field development. HPAM molecule would enter high water saturation formation preferentially. Under the action of hydrogen bond, one end of HPAM molecule would stick to formation surface washed by water. The rest part of HPAM molecule that extends in water acts as an obstacle to mobility of water. So the water phase permeability would reduce. Although HPAM can also generate resistance of oil flow, it can provide a layer of liquid film as lubricating film to reduce oil flow resistance.

(c) Alcohol

YRJ, a kind of alcohol<sup>[11, 12]</sup>, could adhere to the surface of cement powder or exist in pores of cement stone in complex form. In any case, YRJ could adsorb positively charged ions such as calcium ion. In this way, part of cement powder will carry electrostatic charge.

Polarization would occur on polar fluid (water). Dipole moment of water molecule would increase, which also leads the increase of water flow resistance when water goes through pores. However, electrostatic charge has smaller influence on non-polar fluid (oil). In addition, adsorption of YRJ on surface of cement stone could change the surface from water wet to oil wet, increase the interfacial tension between water and pore of cement stone and further increase the water flow resistance.

In this experiment, SP-80, HPAM and YRJ were used as phase-permeability enhancing agent. In order to compare their water plugging and lipophilic property, the oil and water phase permeability of cement stone were measured. The ratio of oil phase permeability to water phase permeability<sup>[5]</sup> ( $K_o/K_w$ ) was used as selecting indicator. The bigger  $K_o/K_w$ , the stronger selective permeability it has.

**3.3.2 Influence of Phase-Permeability Enhancing Agent on Cement Stone Properties**

$K_o/K_w$  was obtained after oil and water phase permeability tests at 60 °C, 70 °C, 80 °C, 90 °C. The details are shown in Table 4. HPAM was ruled out because cement slurry mixed with HPAM has high initial consistency (up to 37 Bc) and low fluidity (13.2 cm) and is not suitable for cementing job.

**Table 4**  
**Test Result of Phase-Permeability Enhancing Agent for Selecting**

Temperature	Parameters	Phase-permeability enhancing agent			
		N/P	0.6% SP-80	3.0% YRJ	3.0% YRJ + 0.6% SP-80
60 °C	$K_o, 10^{-3} \mu m^2$	0.55	0.41	6.88	2.80
	$K_w, 10^{-3} \mu m^2$	0.13	0.06	0.70	0.48
	$K_o/K_w$	4.26	7.38	9.72	5.83
70 °C	$K_o, 10^{-3} \mu m^2$	1.10	0.53	10.08	4.80
	$K_w, 10^{-3} \mu m^2$	0.12	0.04	0.64	0.37
	$K_o/K_w$	8.94	12.14	17.58	12.97
80 °C	$K_o, 10^{-3} \mu m^2$	1.32	0.75	12.74	6.03
	$K_w, 10^{-3} \mu m^2$	0.11	0.04	0.52	0.30
	$K_o/K_w$	11.54	17.93	26.64	20.1
90 °C	$K_o, 10^{-3} \mu m^2$	1.99	0.88	17.98	8.07
	$K_w, 10^{-3} \mu m^2$	0.11	0.03	0.48	0.28
	$K_o/K_w$	18.97	25.68	37.18	28.82

Note. "N/P" means no phase-permeability enhancing agent.

As seen from Table 4, the water-phase permeability of cement stone mixed with SP-80 decreases significantly; however the oil-phase permeability also reduces compared with that of cement stone without SP-80. It is understood that SP-80 could reduce water-phase permeability, but when kerosene flows through cement pores, small droplets of water in oil type, which could increase kerosene flow

resistance, are formed because SP-80 makes emulsification occur between kerosene and free water in cement. Cement stone containing YRJ has highest value of  $K_o/K_w$ . That means cement stone mixed with YRJ possesses the strongest selective permeability to oil and water.

Test results of cement stone compressive strength after displacement are shown in Table 5.

**Table 5**  
**Compressive Strength of Cement Stone**

Phase-permeability enhancing agent	Compressive strength of cement stone after water displacement @ 90 °C, MPa	Compressive strength of cement stone after oil displacement @ 90 °C, MPa
N/P	15.51	12.27
0.6% SP-80	15.53	11.25
3.0% YRJ	13.90	11.06
3.0% YRJ + 0.6% SP-80	14.21	10.80

Note. "N/P" means no phase-permeability enhancing agent.

As Table 5 shows, the compressive strength of cement stone mixed with YRJ after oil or water displacement is slightly lower than that of cement stone with SP-80 or no YRJ. That means YRJ has only marginal effects on compressive strength. In addition, based on previous experiments, YRJ also has good pore-connecting nature. So YRJ is selected as the pore-connecting agent and phase permeability enhancing agent.

## CONCLUSIONS

(a) Oil-soluble cement system, composed of pore-enhancing grains, pore-connecting agent and phase-permeability enhancing agent, is a new cement system with a strong selective permeability to oil and water.

(b) Based on the theoretical analysis, the pore-enhancing grains-ZKJ-1 which can broaden the pore obviously, was experimentally optimized from many kinds of natural oil soluble organics. In order to interconnect the pores of the cement stones and enhance the selective permeability of the cement stone to oil and water, the YRJ was optimized as the pore-connecting agent and phase-permeability enhancing agent by means of theory analysis and extensive experiments.

(c) Cement stone with three basic agents not only has a high value of oil phase permeability, but also possesses a higher compressive strength.

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