Study on Mechanical Property and Blocking Test of Multistage Resin Composite Ductile Cement Slurry System

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Abstract: In view of the traditional inorganic cement-based material hydration shrinkage caused by the impact of performance defects range of application, applying the principle of doping dispersion and compact packing, a multistage resin composite ductile cement slurry system with different temperatures was constructed. The compatibility, thickening and mechanical evaluation tests were carried out to optimize the resin type, doping amount and method. The results show that the thickening of the composite system is controllable, and the addition of resin is beneficial to improve the elastic deformation, whereas easy to pile up if adding extra amounts, and the compressive strength reduced. At the same time, adding resin first is beneficial to disperse in the cement gap. Compared with cement-based materials, the compressive strength increased 7% to 13%, the elastic modulus decreased 10% to 15%, and the deformation capacity increased 14% to 22%. Field test shows that it has good sealing performance and remarkable effect of oil increasing and dewatering, which provides a new technical means for controlling casing leakage and ensuring its normal production.

1. Introduction
As the old oil field enters the stage of high water cut, high recovery and high cost, water flooding is a priority. Water shutoff technology such as suspension and composite inorganic water blocking agent have been used to block water from fracture of limestone reservoir and high permeability zone of sandstone oil well. The mechanism is to block the effluent from fracture and high permeability zone with high strength water blocking agent, to increase production pressure and to find residual oil in tiny rock fracture and low permeability zone. Increasing application of gas injection flooding and construction of underground natural gas storage result in channeling and leakage of casing pipe. The cement ring should be subjected to alternating load during oil recovery or gas injection, which put forwards higher requirements for wellbore integrity. [1-3]

The comprehensive properties of oil well cement stone are important factors affecting cementation. The cement stone is mainly formed by hydration and consolidation of inorganic silicate minerals. Yan Peiyu and others believe that oil well cement stone has defects such as high shrinkage, brittleness and low durability, so gaps and cracks appear in it under strong impact and which results in channeling, leakage and bad cementing quality.[4-5]

In order to improve the toughness and deformation ability of cement stone, elastic materials, latex and fiber are usually added, but this has little effects which improve the toughness at the expense of compressive properties. [6,7] Studies have shown that adding emulsifying resin, epoxy resin and etc
can affect the cement’s hydration process and mechanical property.[8,9] The cement solidify partial without solidifying agent in the low-middle temperature, which in the state of film or gel. In this study the mechanical property of lime slurry is improved by doping organic resin in water shutoff agent so as to meet the requirements of integrity of wellbore.

2. Experiment

2.1. Reagent
G grade cement, lime powder, dispersion agent, early strength agent, retarder, A order phenolic resin crosslinking agent(liquid state), B order phenolic resin crosslinking agent(liquid state), curing agent, C order phenolic resin crosslinking(solid state), C order epoxy resin (solid state), deionized water and so on.

2.2. Apparatus
High energy testing machine, rock tri-axial test system, high temperature and pressure thickening apparatus, cement mixer, channeling test apparatus, steel drum, graduated flask.

2.3. Laboratory methods

2.3.1. Preparation method
Mix the cement, lime powder, resin, early strength agent(retarder) and dispersion agent in proportion and in a certain order, stir them in cement mixer then to prepare complex of resin and lime.

2.3.2. Experiment of thickening
Pour the complex of resin and lime to jar, stir it well and then put it in constant temperature water bath. Record the process of thickening, loss of fluidity and final set. Calibrate the thickening apparatus, pour the complex of resin and lime to sample container, install potentiometer, put the sample container in thickening apparatus water bath, start the motor for thickening, set thickening temperature and record the consistency data of cement paste in different moments.

2.3.3. Mechanical property test
First, pour the cement paste into the mould, put it in water bath maintenance and then mold unloading. In the process of experiment hydraulic universal testing machine applies uniform loading on samples at the rate of 2400(±200)N/s until maximum and then drop, record the data, calculate the compression strength for 24 hours and the elastic modulus.[10]

Tri-axial test: prepare the mandatory sample using the complex of resin and lime slurry according to API standards, test the elastic modulus using GCTS-RTR-1000 tri-axial testing machine, increase the axial load and test axial stress, draw the relationship between stress and strain and get elastic modulus and Poisson’s ratio.

Formulary:

\[ C_s = \frac{M_l}{\pi r^2}, \quad \mu = \frac{B \sigma_3 - \sigma_1}{\sigma_3 (2B - 1) - \sigma_1} \]

\[ E = \frac{(\sigma_1 - 2 \mu \sigma_3)}{\varepsilon_1}, \quad B = \frac{\varepsilon_3}{\varepsilon_1}. \]

\( C_s \) —compression strength, MPa; \( M_l \) —maximum load, N; \( r \) —bottom radius of sample, m. \( E \) —elastic modulus, GPa; \( \sigma_1 \) —axial stress of sample, MPa; \( \sigma_3 \) —pressure on the side, MPa; \( \varepsilon_1 \) —axial strain, \%; \( \varepsilon_3 \) —lateral strain, \%; \( \mu \) —Poisson’s ratio; \( B = \varepsilon_3 / \varepsilon_1 \).
2.3.4. Gas channeling resistance

Pour the sample into channeling testing apparatus, set the temperature and differential pressure, record the differential pressure when the gas channeling happens, evaluate the gas channeling resistance of the sample according to the curve.

3. System development

3.1. Material screening

Screen 4 types of resin to doping with cement lime slurry, record the process of thickening and curing. It shows that in low-middle temperature the A/B resin is incompatible with cement lime slurry, it can’t cure. The C order resin is compatible to cure. In middle-high temperature B and C order resin can cure, but B order resin needs longer time (>5 days). It shows that there are free phenol, formaldehyde in A/B liquid state resin which affect the hydration of calcium silicate deeply but a little on C order resin.

3.2. Component and content optimization

Record the initial concreting time and thickening time of different component and content slurry in different temperature, determine the ratio of water, cement and lime to form the formula. It shows that C order resin doping with cement and lime has a little effect on the thickening with the proportion below 5%, early strength agent and retarder can adjust the thickening time and this is helpful to thickening reaction. B order resin has too long time to lose fluidity and it can’t block the channeling.

Table 1. Thickening test of resin cement lime complex in different temperature

| Temperature °C | Water–cement ratio | Resin types | Early strength agent (retarder) % | Filtrate reducer % | Dispersion agent % | Initial time h | Thickening time |
|----------------|---------------------|-------------|----------------------------------|-------------------|-------------------|---------------|----------------|
| 25–50          | 0.7–0.8             | C phenol aldehyde | 3–5                              | 3                 | 0.3               | 2.5–3.5       | 212–248        |
| 60–80          | 0.6–0.5             | C phenol aldehyde or epoxy resin | 1–3                              | 0.06–0.15         | 1                 | 3.5–4.0       | 260            |
| 90–120         | 0.5                 | B phenol aldehyde | 0.5–2                            | 0.3–0.5           | 3–5               | 6–8           | >360           |

4. Mechanical performance

4.1. Middle-low temperature

Draw the stress-strain curve of cement sample using single axial testing machine (Fig. 1), the curve moves right after adding resin which means the sample’s consistency is improved, but the pressure resistance changed. It shows that the comprehensive mechanical performance is improved comparing to the traditional cement slurry. (Fig. 2) The compression strength increases by 7%–13% in 3% content, elastic modulus decrease by 10–15%, the deformability increases by 14–15%, elastic deformation and plastic deformation occur at the same time.
4.2. High temperature

For the thickening in 110 °C, test the resin and lime stress-strain (Figure 2). Unlike the low-middle temperature, the curve shows obvious characteristics of elastic and plastic deformation. When the stress is <25.4MPa, it is classified into elastic deformation, when the stress is greater than 25.4 MPa less than 36.3 MPa, it reaches the ultimate deformation, the stress-strain curve trends level when it reaches yield stress. The result shows that the pressure bearing performance of resin is improved after doping, the compression strength can raise 14.3% after 2% phenol formaldehyde is added, the elastic modulus reduces by 34.8%, the value of SPN is small which means that it has better gas channeling prevention ability.

Table 2. Mechanical performance of resin and lime emulsion in low temperature

| Temperature °C | Formulation                  | Compression strength MPa | Elastic modulus MPa | Ultimate deformation % |
|---------------|------------------------------|--------------------------|--------------------|------------------------|
| 30            | slurry                       | 18.8                     | 382                | 3.40                   |
|               | +3% C phenol aldehyde        | 20.2                     | 324                | 4.15                   |
|               | +10% C phenol aldehyde       | 16.1                     | 319                | 3.75                   |
|               | +3% C phenol aldehyde        | 21.3                     | 342                | 3.86                   |
|               | +10% C epoxy resin           | 19.0                     | 344                | 3.71                   |

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Table 3. Mechanical performance of the resin and lime complex in high temperature

| Temperature ℃ | Formula                              | Compression strength MPa | Elastic modulus GPa | Poisson ratio μ | SPN  |
|---------------|--------------------------------------|--------------------------|--------------------|-----------------|------|
| 110           | Slurry                               | 50.3                     | 8.9                | 0.4             | —    |
|               | +1%C order phenol formaldehyde       | 52                       | 5.2                | 0.44            | 2.42 |
|               | +2%C order phenol formaldehyde       | 57.5                     | 5.8                | 0.5             | 0.33 |
|               | +3%C order phenol formaldehyde       | 42.9                     | 8.7                | 0.5             | 1.29 |

5. Influencing factors
The type of doping has a major influence on the slurry densification and mechanical property[12-13]. As Fig. 4 and Tab.4 show resin below 5% has little influence on lime cement slurry, the initial setting time with adding resin at last is shorter than doping at first. Adding resin can do good to elastic deformation, but it will reduce the compressive strength when the resin’s content is higher than 3%. The section plane of limestone Fig.3 shows that adding resin at the beginning is better for the dispersing of resin particle in limestone crystal lattice which makes the pore structure compact. Excessive adding of resin or adding at last will make the accumulation of rein which influence the limestone’s mechanical property.

6. Mine experiment
Channeling plugging in 3 wells of sand reservoir and blocking of casing free section in one well were completed successfully.

Example 1: channeling plugging in case Y 11 is one oil well in Fu Shan, produced 100% water because of the channeling in casing pipe. Therefore pour sand to protect the oil layer then perforate in the upper to block the channeling. Daily oil production is from 4.5m3 to 18.9m3, daily water production is from 12.9 m3 to 0.8m3 after blocking.

Example 2: leaking stoppage in free casing pipe J331 is water injection well in J11 fault-block of Bie Guzhuang oil field. Well logging shows that there is leakage in shallow casing pipe, no water sucking in perforation section, the injection water flows into 475-480m. Using circulated injection technique to plugging, the pump pressure is 3–5 MPa, the pressure after plugging is 15MPa.

7. Conclusion

7.1 Chemical resin strengths the limestone crystal lattice, the dispersing of doping resin compacts the plugging agent and improves its mechanical property.

7.2 The type of doping has some effect on plugging agent performance. Adding at the beginning is better for the dispersing of resin between the limestone crystal lattice which has good mechanical property. Excessive adding of resin or adding at last will make the accumulation of rein which influence the limestone’s mechanical property.

7.3 Field test shows that it has good sealing performance and remarkable effect of oil increasing and dewatering, which provides a new technical means for controlling casing leakage and ensuring its normal production.

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