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Research Article

Laboratory Study on a New Composite Plugging Material with High Bearing Strength and High-Temperature Resistance

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The reservoir in the Central Tarim Basin is a typical high-pressure fracture-vuggy carbonate reservoir with high temperatures, which easily leads to drilling fluid losses and the high-temperature carbonate. The conventional lost circulation materials (LCMs) cannot meet the reservoir. To resolve this problem, a rigid particle with high-temperature resistance, high acid solubility, and high rigidity was developed, tested, and evaluated. According to bridge rules of 1/2–2/3 opening degree of formation fracture, the formulation experiments of GZD and other lost circulation materials were conducted and a novel composite lost circulation material (LCM) was completed. Lastly, we investigated the compatibility of LCM and mud in site though compatibility experiment, analyzed the plugging effect of the system for fracture and vuggy by laboratory static lost circulation simulation and evaluation and sand-bed plugging experiments, respectively. The results show that compared with the conventional rigid lost material, the value of high-temperature resistance is more and acid solubility is over 98%. Also, it can work well with other fiber materials (E), variable filling particles (F and G), and superfine filling particles (H) and form a novel plugging material, named MGY-I, whose temperature resistance value is more than 473.15 K, the bearing strength is over than 9 MPa, the mud filtrate invasion depth of sand bed made from coarse particles (10~20 mesh) is only 3.0 cm within 30 minutes, and the invasion depth is less than 1 cm within 30 minutes when the sand bed is made from 80~100 mesh. The optimal concentrations of rigid granule, lignin fiber, elastic particle, and superfine calcium carbonate are 8% (A : B : CD = 2 : 1 : 1), 0.5%, 6%, and 1%, respectively. And, the plugging function of "GZD-rigidity bridge and filling, fiber network and deformable filing" is better exerted on the formation fracture with a high loading capacity and a high-temperature resistance.

1. Introduction

Natural fractures are beneficial for the economical and efficient development of carbonate reservoirs. However, natural fractures also lead to drilling fluid loss and formation damage [1]. Liquid and solid particles penetrate deeply into the reservoir and cause serious damage [2–4]. Particle invasion, phase trapping damage, and rock-fluid incompatibility are the main causes of formation damage [5–7]. These problems are prominent in the Central Tarim Basin. This basin also contains blocks of ultrahigh-temperature reservoirs with temperatures above 453.15 K. These areas lead to the carbonization phenomenon of LCM. Therefore, an efficient technology of lost circulation control and plugging must be used [8–11]. Currently, bridging plugging technology is the most popular method, and hardness, bearing strength, and high temperature are the main factors that lead to lost circulation control in high-temperature fracture-vugg reservoirs [10]. Both fibrous plugging material and rigid particles are used in this technology. Although the fibrous material can form a skeleton, the skeleton is prone to high-pressure destruction [12]. In contrast, rigid particle bridging and plugging results lead to structures with high bearing strengths that are not easily deformed. This technology has many applications. However, the conventional materials used in rigid particle bridging and plugging are easily carbonized at high temperatures, and their hardness is compromised under these conditions. The bearing strength of the plugging layer must be abated. Under these conditions, the plugging layer loses its effectiveness, resulting in losses and formation damage [13]. Many reports describe plugging materials with high-temperature resistance; these include mica, vermiculite, asbestos, and shell [14–16]. There are many reports such as Alsaba et al. [17], Guo et al. [18], Li et al. [19], Wang et al. [20], Guo et al. [21], Karcher et al. [22], and others of other types of circulation material loss. These include adhesive sealing material and consolidated sealing material that provide high-temperature resistance. These materials still have certain limitations when they are used for drilling in fracture-vuggy reservoirs at abnormally high temperatures, such as high-temperature resistance and compressive strength. To resolve the problem of lost circulation under these conditions, we introduce a rigid particle, with high-temperature resistance, high acid solubility, and high rigidity. This particle together with lignin fiber elastic material, SQD-98 and superfine calcium carbonate, as bridging and filling material, produces a novel drilling fluid.

2. Characteristics of Different Kinds of LCM

2.1. GZD. GZD is a rigid temporary plugging particle with a serrated surface (shown in Figure 1) and wide particle size distribution. It is mainly composed of the calcite, its density is 2.8 g/cm^3, and its Mohs hardness is 2.7~3.0. The particle diameter is 0.2~2.0 mm, mainly divided into 4 grades, including A, B, C, and D, as shown in Figure 2. It has the function of bridging and filling and good temperature resistance and can resist temperature over 473.15 K. The acid solubility of GZD is more than 98%, which is beneficial to the later plugging with acidification, and protects the reservoirs.

2.2. Fiber. Fiber is a common kind of LCM. The lignin fiber used in the experiment is an organic fiber obtained by screening, splitting, and chemical and high-temperature treatment of natural timbers. The treatment temperature reaches up to 523.15 K. It has good temperature resistance performance, and it is a nontoxic and harmless green environment-friendly product. Its microscopic physical structure presents strip bending, the sags and crests, and the porous intersection, and it has good water absorption, dispersibility, and flexibility. Compared with the conventional fiber materials, the acid solubility of the modified lignin fiber is over 39%, which is beneficial to removing and the efficient development of the oil fields.

2.3. Calcium Carbonate. As a rigid plugging material, calcium carbonate has been widely used in the oil industry. Its density is 2.6~2.9 g/cm^3, its Mohs hardness is 3.0~3.5, its acid solubility is more than 99%, and it has good protection performance for the reservoirs. The mixing ratio of the calcium carbonate used in the system is 0.019 mm: 0.0021 mm (800 mesh: 1200 mesh) = 1 : 1.

2.4. SQD-98. SQD-98 is an elastic filling particle with a 0.07~4.20 mm particle size. It has good plugging and temperature resistance and is often used in the oil industry as a plugging material. The addition of elastic particles will increase the friction between the plugging zone and the fracture surface because of their elastic deformation, thus improving the stability of the plugging zone.

3. Laboratory Evaluation of Plugging Effect with LCM Combinations

3.1. Experimental Setup. The major apparatus, as shown in Figure 3, consists of two parts: (a) the plugging fracture evaluation device (BTM) and (b) the plugging evaluation device for holes. The apparatus used for optimizing the LCM doses, for static sealing and for the bearing fracture pressure effect, consists of an N_2 cylinder, a vessel, an accumulator, a core holder, and a WLB-100 pump. Hydraulic oil, pressurized by the WLB-100 pump, was used to supply a confining pressure to the core which was made of steel block as shown in Figure 4. The Visual FA model filter tester contains an accumulator, a measuring cylinder, and an N_2 cylinder that supplies the displacement pressure for the filter tester.

3.2. Experiment Design and Evaluation Method. The objective of laboratory experiments conducted in this study is to test the characteristics of GZD, investigate and determine the optimal combination of GZD and other LCM, and evaluate the compatibility of LCM and field drilling fluid and the effectiveness of drilling fluid for fractures and holes. The integral experiment was divided into four parts shown as follows.

3.2.1. Evaluation of Characteristics of GZD. This evaluation includes three parts, which is to investigate performance of GZD, including thermal ability, temperature abrasiveness, and acid solubility. Firstly, thermal ability of GZD was investigated with DSC (differential scanning calorimeter). Secondly, the high-temperature abrasiveness of GZD was measured with the traditional experimental method, rolling recovery. The former experiments were carried out by the law of DSC. The latter experimental steps were described as follows: (1) weigh a certain quality sample, 30 g, and mix into basic mud, 2% bentonite + clean water + 0.2% NaOH + 0.3% Na_2CO_3; (2) put the mud into the aging kettle and roll for 16 h at 473.15 K, respectively; (3) remove and wash residual samples and then dry at 375.15 K to constant weight; (4) calculate the wear rate, η, according to formula $\eta = (M_1 - M_2) \div M_1 \times 100\%$. M_1 is the initial weight, and M_2 is the residual mass. Finally, the acid solubility of GZD was tested and investigated. The specific experiment steps were shown as follows; (1)make up mud acid with hydrofluoric acid and hydrochloric acid of certain proportions on the basis of the formula, 2% HF + 10% HCl; (2) samples of a certain quality (shown in Table 1), GZD and $CaCO_3$, were weighed and put into the mud acid to dissolve at 353.15 K for 2 hours; (3) filter and wash the residue with a filter and distilled water; (4) firing the residue and filter to constant

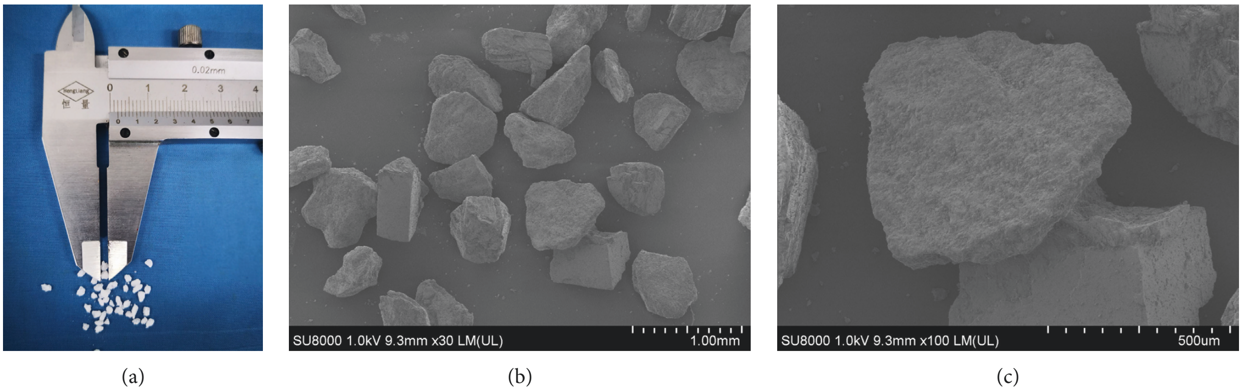


Figure 1: Morphology characteristics of GZD.

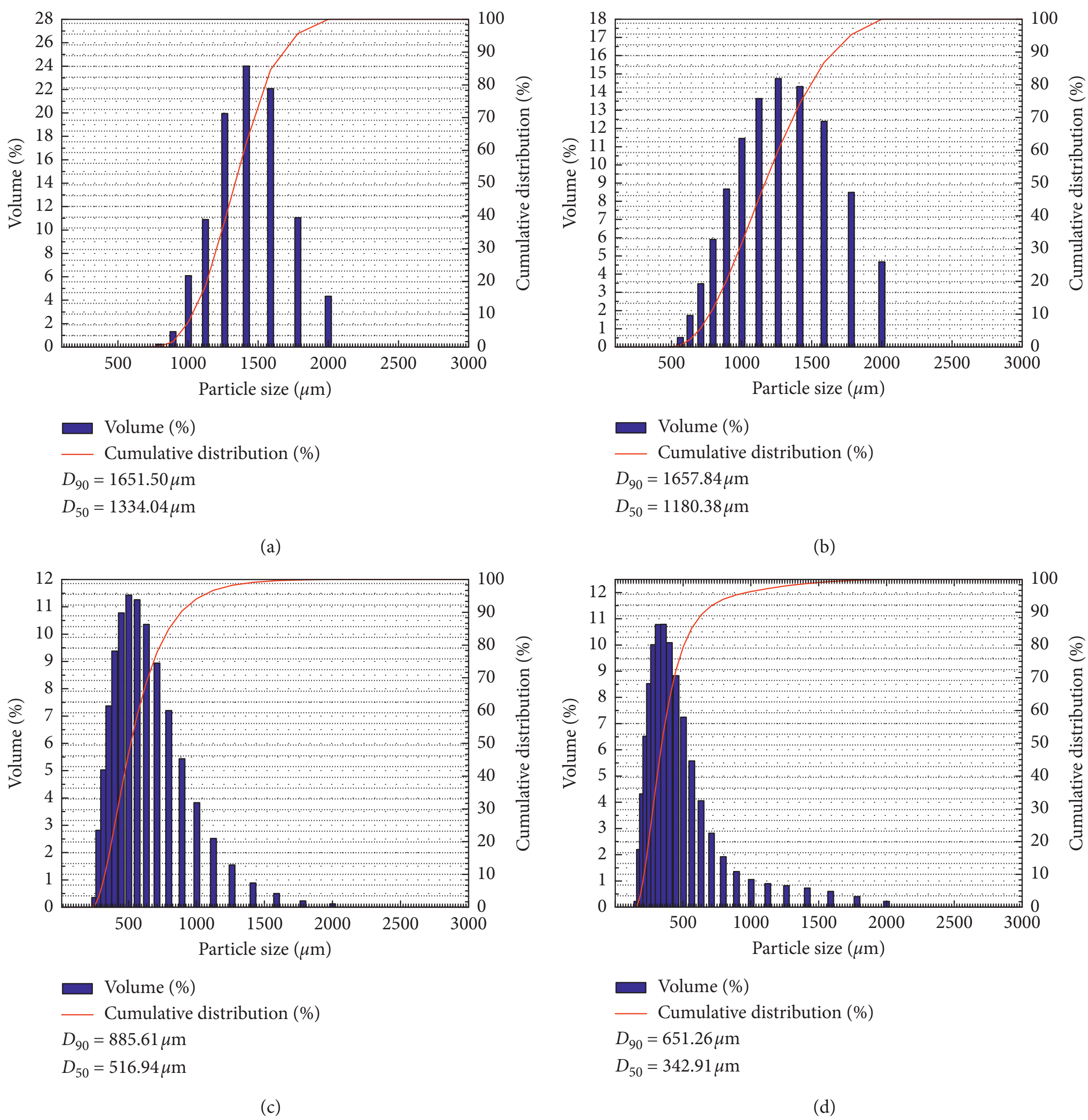


Figure 2: Distribution diagram of particle size.

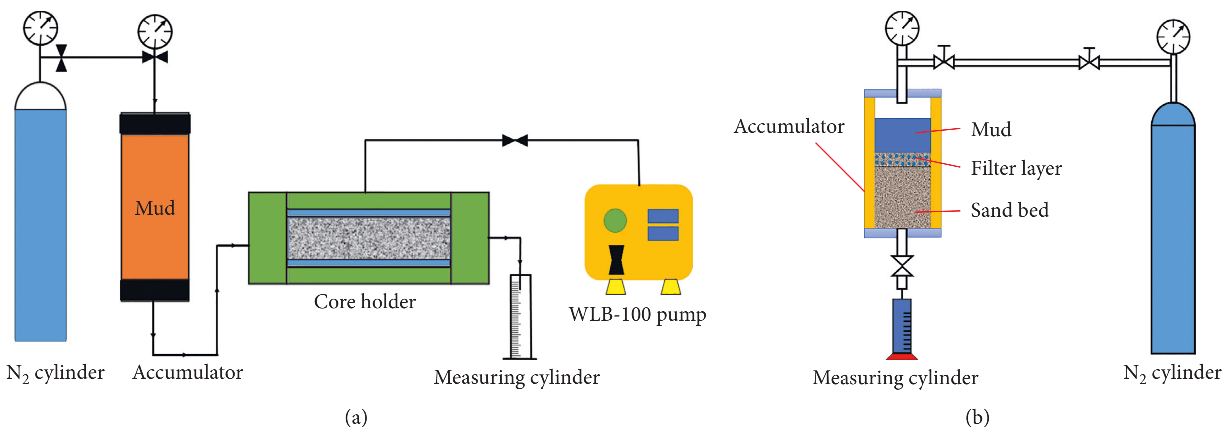


FIGURE 3: Schematic diagram of experimental setup. (a) The equipment of static pressure. (b) Visual FA model filter tester.

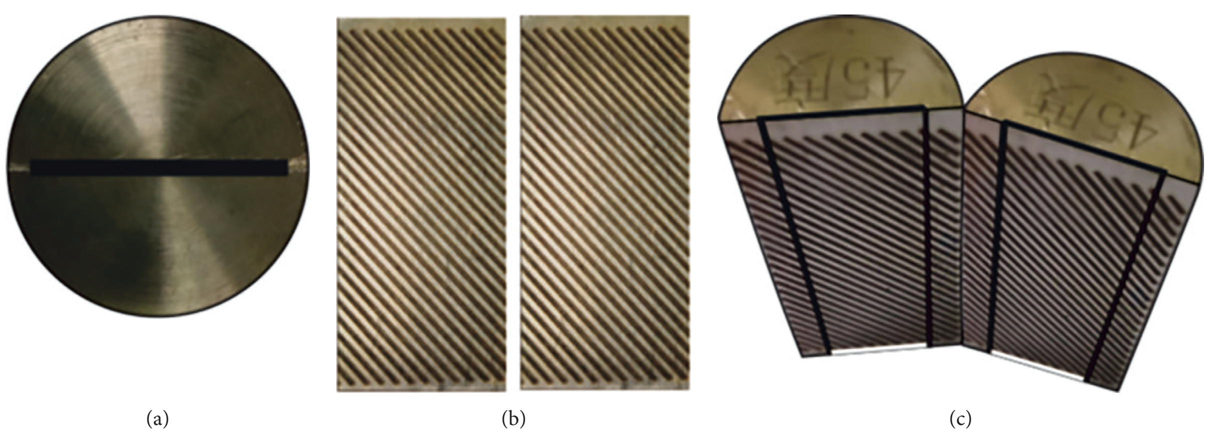


FIGURE 4: Diagram of fracture core.

TABLE 1: Measured acid solubility for the GZD.

Sample name	Mass (g)	Filter paper mass (g)	Paper and residual mass (g)	Acid solubility (%)	Average value (%)
GZD	5.00	0.81	0.92	97.83	98.1
	5.01	0.79	0.83	98.37	
$CaCO_3$	5.00	0.80	0.85	99.00	98.70
	4.99	0.81	0.89	98.40	

weight at 773.15 K and weigh. Meanwhile, the DSC value and rolling recovery of walnut shell were tested and compared with GZD.

3.2.2. Optimization Experiment of Materials. The objective of the laboratory experiments conducted in this study is to determine the optimal combination and concentration of GZD and other LCMs for fractures and vug on the basis of characteristics evaluation of GZD. Two key indexes were adopted to evaluate the plugging effect of LCM in this study: loss pressure and loss volume. Loss pressure refers to pressure a fracture-plugging zone can withstand before it becomes unstable and breaks. The loss volume under special pressure is taken as loss volume. The test procedures are as follows: (1) the basic mud added with special LCMs was injected into the fluid container, the initial pressure point was set at 0 MPa for 1 min, and then, the fluid outlet was opened; (2) the basic mud loss and fracture plugging process was simulated by applying the pressure gradient of 0.5 MPa; (3) the pressure at which the plugging zone broke was recorded and the previous pressure point was taken as the maximum plugging pressure. The basic mud volume at each pressure point was measured, and the cumulative loss volume when the maximum plugging pressure was reached was taken as the total loss volume before sealing.

3.2.3. Evaluation of Compatibility of LCMs and Field Drilling Fluid. The objective of the laboratory experiments conducted in this study is to investigate the compatibility of LCMs and field drilling fluid and analyze the effect of LCMs

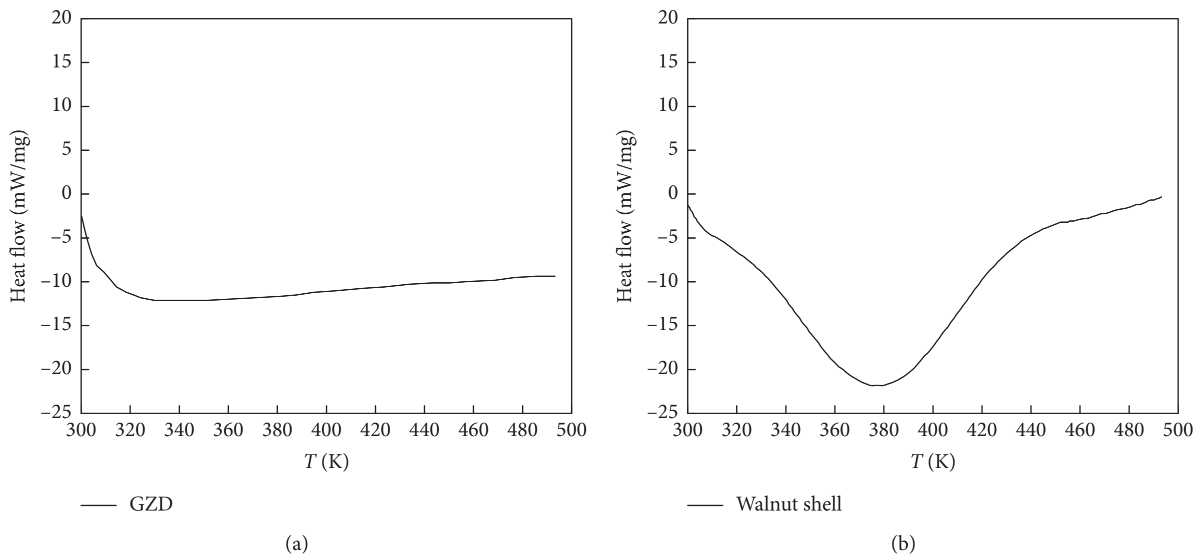


Figure 5: DSC results of GZD and walnut shell.

Table 2: Measured temperature resistance for the GZD.

Sample name	No	Mass loss rate η (%)
Walnut shell	1#	32.97
	2#	31.93
	3#	32.03
	Average value	32.30
GZD	1#	4.50
	2#	4.57
	3#	4.47
	Average value	4.51
Appearance	 Unheated (walnut shell); Heated (walnut shell); Unheated (GZD); Heated (GZD)	
Remark	Distilled water + 0.2% NaOH + 0.3% Na_2CO_3; the same below; 473.15 K, 16 h	

in the performance of field drilling fluid. The test procedures are as follows: the characterization parameters were simulated before and after LCMs were added and the parameters were compared to investigate the compatibility.

3.2.4. Evaluation of Plugging Experiments. On the basis of (1), (2), and (3), a novel plugging drilling fluid was developed. The objective of the laboratory experiments includes two parts, fractures and core plugging, to investigate the plugging effectiveness of drilling fluid for fractures and holes, respectively. The test procedures are as follows: (1) the drilling fluid added with special LCMs was injected into the fluid container to investigate the plugging effectiveness of drilling fluid for fractures; (2) the drilling fluid added with special LCMs was injected into the fluid container filled with different debris with particle size to the container to investigate the effectiveness of drilling fluid for holes. In the same way, three key indexes, maximum plugging pressure, loss volume, and total loss volume before sealing, were adopted to evaluate the plugging effectiveness of drilling fluid for fractures and holes.

4. Results and Discussion

4.1. Performance of GZD. The production capacity of reservoirs declines and formation damage occurs when plugging layers are not promptly formed and removed [23, 24].

In order to prevent loss of drilling fluid and reduce the formation damage effectively, it is the preferred option to use the engineering plugging materials with excellent performance as plugging materials. In the paper, a rigid temporary plugging particle, GZD, was recommended and its relevant features and walnut shell were evaluated. The results are shown in Figure 5 and Table 2.

Experimental results in Figure 5 show that the DSC endothermal value first increase then remains essentially unchanged with the increase of temperature, indicating that GZD has good thermal stability and can keep its original characteristics under high temperature. However, the distribution range of DSC value of walnut shell is more fluctuate, which indicates that walnut shell is extremely unstable when the temperature is raised.

The high-temperature abrasiveness reflects the strength and temperature resistance of material and decides the loading capacity and the plugging capacity of the plugging zone at high temperature. Therefore, we tested the abrasiveness at 453.15 K, and the results are shown in Table 1.

According to a comparison analysis of Table 1, we can see that η_{shell} is greater than the η_{GZD}, indicating that GZD has greater high-temperature resistance than walnut shell and almost no thermal decomposition at 473.15 K. Walnut shell undergoes a color change, from rufous to dark brown. This change indicates high-temperature gelatinization. In contrast, GZD did not change color and did not undergo pyrolysis or high-temperature degradation. GZD has good high-temperature resistance, so it can be used as lost circulation material in high-temperature reservoirs. The results maintain consistency with the experimental results in Figure 5.

The production capacity of reservoirs declines and formation damage occurs when plugging layers are not promptly removed [23, 24]. Acid treatment is an important technology for removing plugging material in oilfields. Therefore, we investigated the solubility of GZD in acid and contrasted with the acid solubilization of $CaCO_3$. The results are shown in Table 2.

Experimental results in Table 2 show that GZD has high acid solubility and the average value is over 98.1% and close to the value of calcium carbonate (98.7%), which is good for the technology of removing plugging material and formation protection.

4.2. Laboratory Evaluation of Plugging with LCM Combination. GZD is a rigid temporary plugging particle with a serrated surface and a wide particle size distribution. We need to determine the optional proportion of different grain diameters, A, B, C, and D. The concentration of different grain diameters of GZD combinations is shown in Figure 6.

Experimental results in Figure 6 show that GZD, A, B, C, and D, can form a plugging zone in a smaller pressure and has a minimum volume loss of 10 mL/3 MPa; the loss volume from 200 mL/0 MPa to 10 mL/3 MPa represents a loss of 95%, indicating that the plugging effect can be improved with the combination of GZD with different grain diameters A, B, C, and D of 95%. But, Figure 6 shows that this single plugging material cannot form a plugging zone with high bearing strength, which is consistent with the basic theory [8, 11].

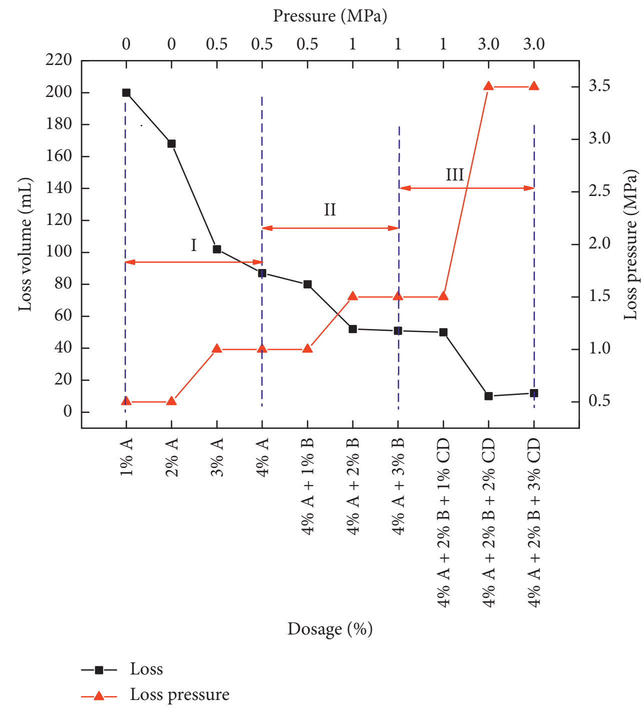


Figure 6: Evaluation of GZD.

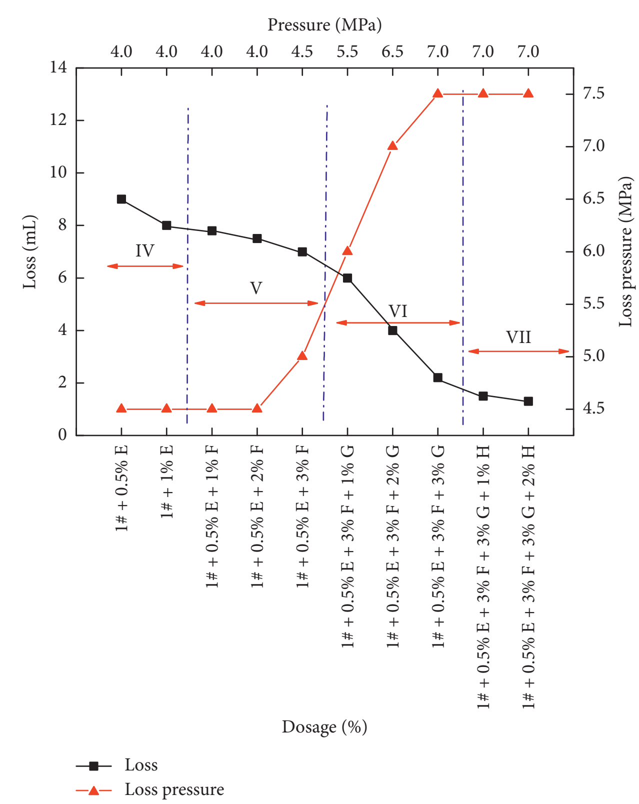


Figure 7: Optimization of GZD, fiber, SQD-98, and $CaCO_3$.

Figure 7 shows the results of adding other plugging materials: fiber, SQD-98, and 1200 mesh $CaCO_3$ concurrent with GZD, which leads clearly to volume reduction and increasingly significant pressure loss.

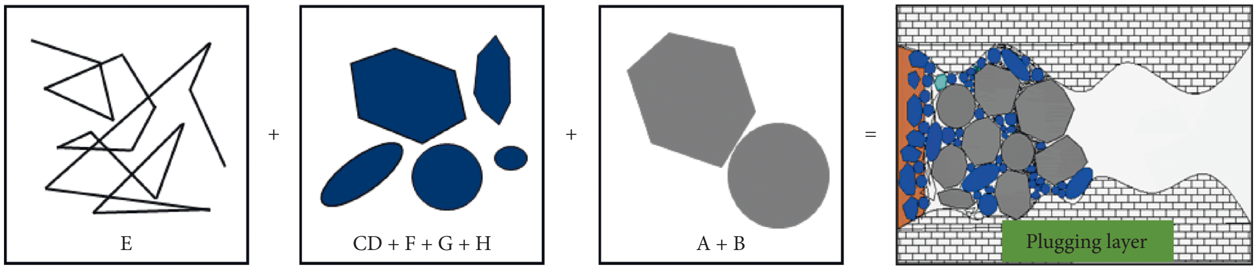


FIGURE 8: Schematic diagram of synergetic of A, B, CD, E, F, G, and D.

The experimental results of Figure 7 show that the loss volume decreases from 7.5 mL to 1.5 mL, an 80% reduction, and the loss pressure increases from 5.0 MPa to 7.5 MPa, a 40% increase. These results can be explained in terms of the SQD-98 fiber, which is a deformable material, and $CaCO_3$ as a superfine filter. When this combination is squeezed into fractures and small cracks of the skeleton, the pulling, filling, and bonding makes the plugging zone more solid and dense [25].

The deformable particles of SQD-98 have strength and elasticity that display dual functions of expansion and compaction under the pressure difference. These properties improve the stability of the plugging layer and produce a good sealing effect [26].

The above results imply that the major rigid granule, GZD, has good bridging and filling action and can work well with other plugging materials. The schematic diagram of four types of synergetic plugging materials is shown in Figure 8.

The experimental results in Figures 6 and 7 indicate that the combination of the rigid granule (GZD), fiber, elastic particle (SQD-98), and superfine filling particle (CaCO3) can create a synergistic effect to achieve the optimal value of the maximum plugging pressure and the minimum loss volume before sealing. For a 3 mm fracture, the optimal concentration of the rigid granule GZD fiber, elastic particle SQD-98, and the superfine filling particle $CaCO_3$ is 8% (A : B : CD = 2 : 1 : 1, CD (C : D = 1 : 1)), 0.5%, 6%, and 2%, respectively. The experimental results are described in detail in the next sections.

4.3. Compatibility Experiment Results. According to the proportion optimized in Section 4.2, add the materials into the field drilling fluid and test the rheology and the filter loss at the room temperature and high temperature. By comparing and analyzing the values, we can evaluate the compatibility of lost circulation materials and drilling fluid. The experimental results are shown in Table 3.

The plugging materials have improved compatibility with field drilling fluid. Plugging materials do not affect the rheological property of drilling fluid, but they do improve the filtration property and they reduce the volume loss. From these results, we developed a new type of plugging-while-drilling fluid with the formula: the mud + 8%~10% GZD (A : B : CD = 2 : 1 : 1, CD (C : D = 1 : 1)) + 0.5%~1% fiber + 5%~6% SQD-98 + 1%~2% $CaCO_3$.

4.4. Plugging Fracture and Vug Adaptability. On the basis of 4.1, 4.2, and 4.3, a novel plugging drilling fluid was developed and through the static sealing and bearing pressure experiments and cutting bed experiments, we analyzed the plugging effects of the lost circulation curing drilling fluid for the fracture and vuggy. In this section, two key indexes are proposed for efficiency evaluation of fracture plugging and one key index is proposed for holes plugging evaluation. There is the loss volume before sealing, pressure, and invasion depth of mud for the sand bed. The sand bed has five ranks, which are made of different particle size debris, and the method is as follows: (1) the debris is divided into five levels: 10–20 mesh, 20–40 mesh, 40–60 mesh, 60–80 mesh, and 80–100 mesh; (2) putting the debris into accumulators to form sand bed with different bore diameters.

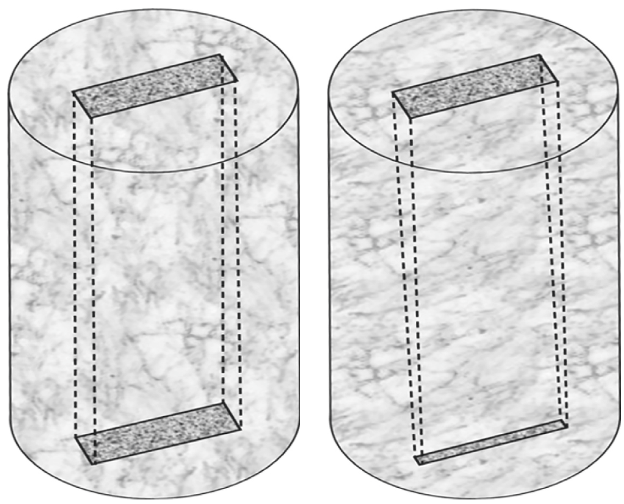
For optimum results, the LCM is added into the oilfield mud to develop drilling fluid and to analyze the resulting plugging effect for fracture. In our experiments, two cores were used: one had a parallel fracture and the other was a wedge-shaped fracture. Figure 9 shows the schematic diagram. Figure 10 shows the experimental results.

The results in Figure 10(a) indicates that the loss volume increases at first and then decreases as the pressure increases. The pressure finally falls to 0. This result is because plugging materials form an effective plugging layer at zero pressure difference or low dropout. Once the plugging layer is formed, the loss volume is reduced, and the loss pressure is gradually increased as shown in Figure 10(a). This figure also shows that loss stops when the pressure is greater than 7 MPa after more than 19 min, which means that a compact and bearing strength plugging layer is formed in a relatively short period. Also, the total loss volume is less than 14 mL in 30 min, and the system maintained zero loss when the pressure was greater than 10 MPa.

The results in Figure 10(b) are consistent with those in Figure 10(a): the loss volume first increases and then decreases as time goes on, and with increasing pressure, the loss volume finally decreases to 0. The bearing strength is greater than 10 MPa. In contrast, the wedge-shaped fracture (2×1.5 mm, 2×2 mm) is faster in forming the plugging layer and the total loss volume is less than the parallel fracture (2×2 mm), less than 13.5 mL. We attribute this to the flow resistance in the wedge fracture being larger than that in the parallel fracture so that the flow resistance is beneficial to the formation of a plugging layer; and therefore, the formation of the plugging layer is more rapid.

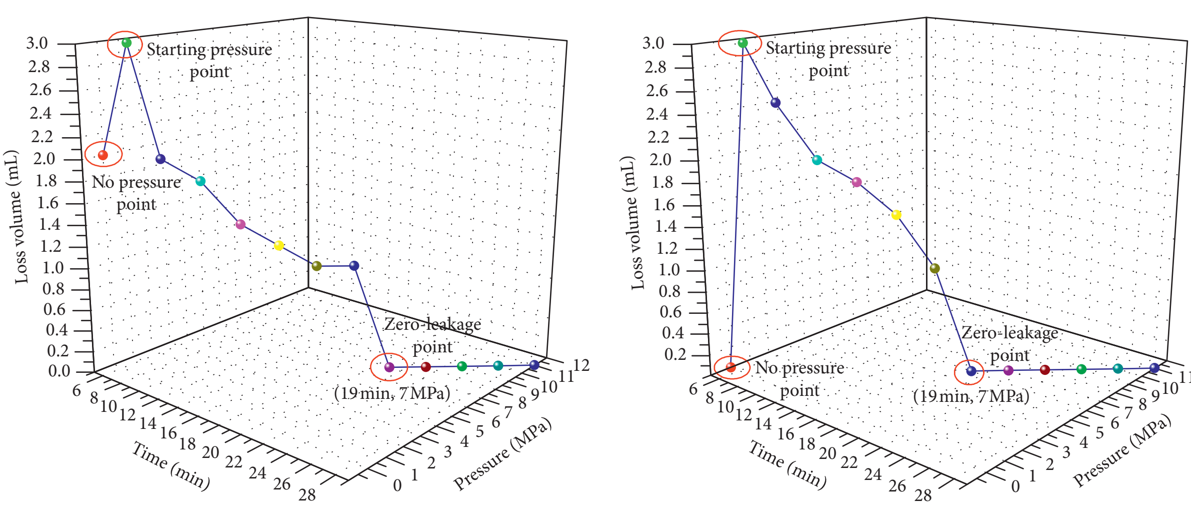
Table 3: Compatibility evaluation of plug materials and drilling fluid.

No	Formula	ρ (g/cm^3)	AV (mPa·s)	PV (mPa·s)	YP (Pa)	Gel$_{10\,s}$/gel$_{10\,min}$ (Pa/Pa)	FL$_{API}$ (mL)
a	Drilling	1.80	65	57	8	1.5/2.0	3.8
	Drilling and plug materials	1.86	75	64	11	2.0/2.5	3.6
b	Drilling	2.00	88	76	12	2.0/2.6	3.6
	Drilling and plug materials	2.05	97	80	16	2.0/3.0	3.2
Remark	a: at room temperature (25°C), 16 h; b: at high temperature (200°C), 16 h						



(a) (b)

Figure 9: Schematic diagram of core. (a) Parallel fracture (2 × 2 mm). (b) Wedged-shape fracture (2 × 1.5 mm).



(a) (b)

Figure 10: Evaluation of plugging effect of fracture. (a) 2 × 2 mm. (b) 2 × 1.5 mm.

There are not only fractures in the reservoir but also a large number of vugs. The results are shown in Figure 11.

Figure 11 shows the results of plugging drilling fluid named SXM-II for vugs. The mud filtrate invasion depth is increased when the pore-throat radius of the mesh of debris decreases. The plugging drilling fluid presents a larger plugging effect when compared with the mud. Under the same conditions, the invasion depth of the sand bed made with debris with a particle size of 10–20 mesh reduces the invasion depth from a whole loss in 1 min to the value of 2.5 cm in 30 min. In the same period, with the decrease of particle size, from 20–40 mesh to 80–100 mesh, the mud filtrate invasion depth is reduced from 12 cm, 7.2 cm, 2.6 cm, and 1.7 cm to 1.2 cm, 1 cm, 0.8 cm, and 0.6 cm, respectively.

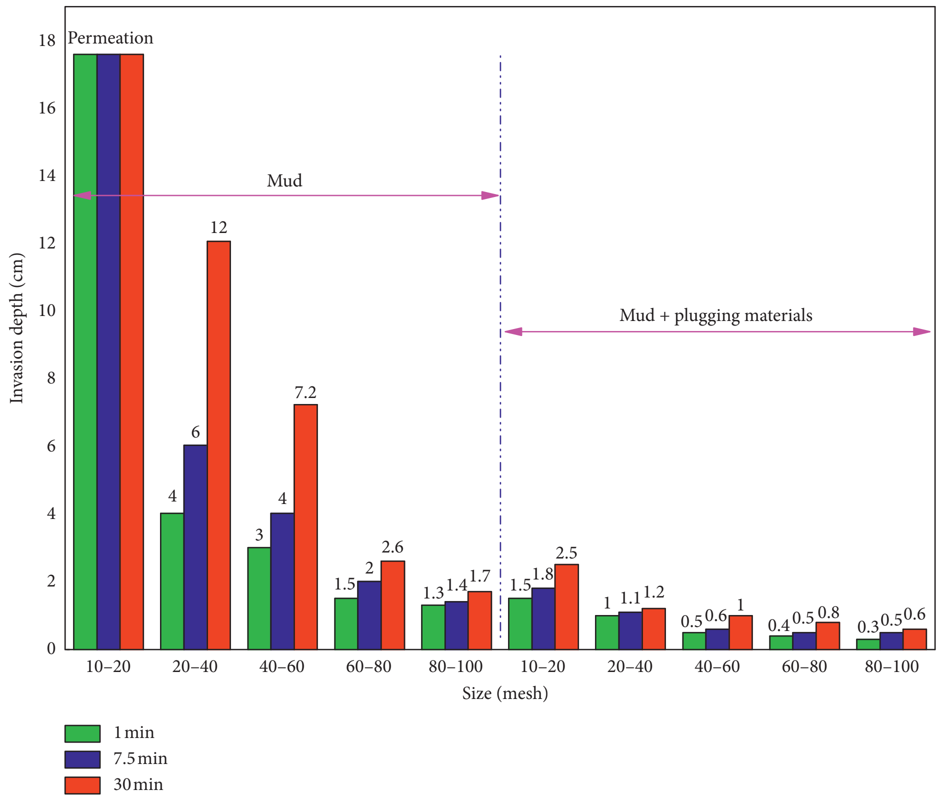


Figure 11: Evaluation of plugging effect of vug.

Also, the depths are greatly reduced, showing that the system has great plugging ability for the vuggy reservoir. In contrast to the plugging effect for a sand bed with the smaller pore size, the plugging drilling fluid has better sealing effect for a sand bed with larger pore size. A sand bed with larger pore size is formed by larger particles. As the materials enter the holes, they form a plugging layer with lower permeability. This layer stops the liquid invasion, and the layer is protected from formation damage. Meanwhile, it can effectively avoid the formation damage resulting in a permeable loss.

In conclusion, the plugging drilling fluid has a two-fold plugging effect. This fluid not only seals fractures but also plugs holes. This drilling fluid can also be used when drilling a fracture-vuggy reservoir.

5. Conclusion

(1) The rigid lost circulation material GZD with high-temperature resistance, high-temperature abrasiveness, and high abrasion performance was developed.

(2) GZD can form a new type of composite lost circulation material with high-temperature resistance and high-pressure bearing with lignin fiber, elastic lost circulation material SQD-98, and calcium carbonate. The main composition and matching proportion are shown as follows: (8% to 10%) GZD (A : B : CD = 2 : 1 : 1, CD (C : D = 1 : 1)) + (0.5% to 1%) lignin fiber + (6% to 8%) SQD-98 (medium: coarse = 1 : 1) + (1% to 2%) calcium carbonate (0.0150 mm: 0.0021 mm (800 mesh: 1200 mesh) = 1 : 1).

(3) The lost circulation material has a good compatibility with the current drilling fluid system in the Central Tarim Basin, and the formed plugging drilling fluid can resist a temperature more than 180°C and a density of 1.80 g/cm^3 or more, which meets the drilling demand on the high-temperature and high-pressure wells.

(4) The plugging drilling fluid system has good plugging effect on fractures and vugs. The plugging and pressure bearing of fracture is above 9 MPa, and the cumulative leakage loss is reduced to 13.4 mL. The cumulative invasion depth of the large-diameter rock debris sand bed is only 2.5 cm in 30 min, and the cumulative intrusion amount of small-diameter rock debris sand bed is less than 1.0 cm in 30 min.

Data Availability

The original and processed data used to support the findings of this study are currently under embargo while the research findings are commercialized. Requests for data, 12 months after publication of this article, will be considered by the corresponding author. If readers have any question for the data, they could contact the author through e-mail sxm310426@126.com.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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