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Development and Performance Evaluation of Novel Solid-Free Epoxy Resin System for Remediation of Sustained Casing Pressure

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Abstract: Sustained casing pressure (SCP) is a common problem during an entire life span of oil and gas wells. When conventional methods are deployed to resolve the issue, the methods seem to have some setbacks by being unable to seal microcracks in the wellbores. A new solid-free resin sealant was developed by increasing the amount of active hydrogen in the curing agent. This low-temperature cure results in low viscosity, excellent mechanical properties, and chemical stability. The experimental results show that the resin sealant can be used at temperatures ranging from 20 to 50 °C, and the curing time can be controlled within 0.25 to 20 h by increasing the curing agent content. The viscosity of the resin is reduced from 35.7 Pa·s to less than 0.065 Pa·s with the addition of the viscosity reducer, which can greatly promote resin penetration into the microcracks of the cement sheath. After 24 h of curing, the compressive strength can reach 55 Mpa, which is significantly higher than conventional cement. With an increase in the viscosity reducer content from 0% to 25%, the elongation of the sealant demonstrates a remarkable rise, ranging from 1.9% to 18%. The cement with 20% resin caused a significant decrease in permeability by 46.3% as compared to conventional cement. Additionally, the sealing pressure attained an impressive value of 14.7 Mpa. The solid-free resin sealant is miscible with cement slurry, which improves cement tightness, reduces permeability, and improves wellbore annulus sealing ability.

Keywords: curing agent; viscosity reducer; compressive strength; solid-free resin; break elongation

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1. Introduction

Sustained casing pressure (SCP) is a common problem that can occur throughout the lifetime of the oil and gas wells, but it is frequently discovered in the later stages [1–3]. According to the US Mineral Management Service, SCP affects over 8000 wells in the Gulf of Mexico [4]. SCP has been reported from 63.7% of shale gas wells in the Sichuan Basin after staged fracturing [5]. SCP is commonly caused by tubing, casing, well-head leaks, poor primary cement, or damage to primary cement after setting [6]. Leaks can occur due to a poor thread connection, corrosion, thermal-stress cracking, or mechanical rupture of the inner string [7,8]. Furthermore, large-scale hydraulic fracturing is required to stimulate unconventional reservoirs, which has the potential to weaken the casing cement bond or develop cracks and fissures in the annular cement [9]. SCP is an ongoing safety hazard that, if not controlled, can cause serious and immediate harm or damage to human life, the marine and coastal environment, and property [10]. Therefore, there is an urgent need to address SCP issues and maintain wellbore integrity.

Squeezing cement is often the solution to SCP. Cement is forced into cement cavities and/or between the cement and the surrounding environment [11,12]. Many materials can be used to seal the fluid pathways that are created in the wellbores by SCP; traditional Portland cement, microfine cement, ultrafine cement, polymer gels, and polymer resins are some example materials [13]. Conventional oil field cement is injected to repair the primary cement if the aperture size is more than 400 µm [14]. Microfine and ultrafine cement can handle an aperture size less than 400 µm but higher than 120 µm in width [15]. However, they are limited by the vulnerability of the cement's thickening time during the operation, and it also requires casing perforation in general [16]. Polymer gels are polymer-crosslinker mixtures that can be combined and optimized to transition from liquids at the surface to semisolids in situ [17]. Pressure, temperature, salinity, or acidity can all activate polymerization. However, the development of polymer gels is limited by mechanical strength and bonding properties [13]. Polymer resins may be used as alternatives in cement remediation to overcome the limitations of conventional cement, particularly in very tight gaps [18,19]. Resins can also be mixed with cement to increase the overall mechanical properties and chemical stability [20,21].

With the advancement of oil and well exploration, increasingly complex working conditions have raised the bar for repairing materials. Polymer resins are mixtures of base resins and hardeners (curing agents) and are superior repairing or cementing agents [22]. They can be pumped deeper into microchannels than cement because it they are solid-free and they have good penetrating properties [23]. Furthermore, polymer resins have high mechanical strength, excellent bonding properties, no by-products during hardening, and little to no shrinkage [24–26]. The resin can effectively fill the cracks and defects on the cement surface, improving the wellbore annulus's tightness and strength. Resins typically have a shorter curing time than cement, enabling the formation of a robust seal to occur more quickly, thereby reducing construction time [27,28]. These characteristics make them most promising materials for good repair [29,30]. However, selecting the appropriate sealant is dependent on the field job itself, so there are no general guidelines or rules to follow in every case. As a result, it is critical to evaluate the properties of resin sealants and the relationship between formulations and properties in a systematic manner.

This research created solid-free resin sealants with low viscosity, low-temperature curing, excellent mechanical properties, and chemical stability. The low-temperature curing process of resins is improved by increasing the number of active hydrogens in the curing agents. In addition, an appropriate viscosity reducer 660 was added to improve the performance of the resins. The sealant system has excellent mechanical properties, ensuring safe development and extending the service life of oil and gas wells.

2. Materials and Methods

2.1. Materials

Epoxy resin (Bisphenol-A E-51) has the characteristics of low viscosity, high epoxy value, and stable performance. The sealant that was used to seal the casing annulus was created by mixing epoxy resin with a viscosity-reducing agent and a curing agent. This sealant has good injectability and low temperature curing ability. In this study, the viscosity reducer was chosen from three widely used commercial short-chain hydrocarbyl glycidyl ethers: butyl glycidyl ether (660), ethylene glycol diglycidyl ether (669), and phenyl glycidyl ether (690), all of which were purchased from Zhenjiang Danbao Resin Co., Ltd. (Zhenjiang, China).

The parameters of the epoxy resin are shown in Table 1. In addition, the epoxy resin was acrylate modified to improve its mechanical properties and durability. In the beginning, the weighed epoxy resin E51 and the mixture of n-butanol and ethylene glycol butyl ether were placed in a 250 mL four-necked flask equipped with a condenser and dropping funnel, stirred, and completely dissolved at 100 °C. The mixture of methacrylic acid, benzoyl peroxide, and diacetone acrylamide was then slowly added in drops over 2 h while stirring. For 4 h, the reaction was kept at 115 °C. Finally, the temperature was reduced to 50 °C, and a dropwise addition of N, N-dimethylethanolamine, and n-butanol was made to neutralize the solution. Emulsification with water produced the modified epoxy resin product.

Table 1. The parameters of epoxy resin (Bisphenol-A E-51).

Epoxy Resin	Parameters			
Bisphenol-A E-51	Viscosity (Pa·s)	Epoxy value (eq/100 g)	Organochlorine (eq/100 g)	Volatile fraction(%)
	<2.5	0.48~0.54	≤0.02	≤2

2.2. Synthesis of the Curing Agent

The highly reactive polyamine low-temperature curing agent was synthesized at 70~90 °C using the thermostatic reflux method, and the experimental devices are depicted in Figure 1. A 500 mL three-necked flask equipped with a condenser tube, thermometer, and mechanical stirrer was filled with phenol and isophorone diamine. This was slowly heated to dissolve the phenol completely. When the temperature reached 80 °C, the weighed paraformaldehyde was added and reacted for a set amount of time at 90 °C. Finally, the product was dehydrated completely at 110 °C under atmospheric pressure, and the resulting red-brown liquid was used as the curing agent.

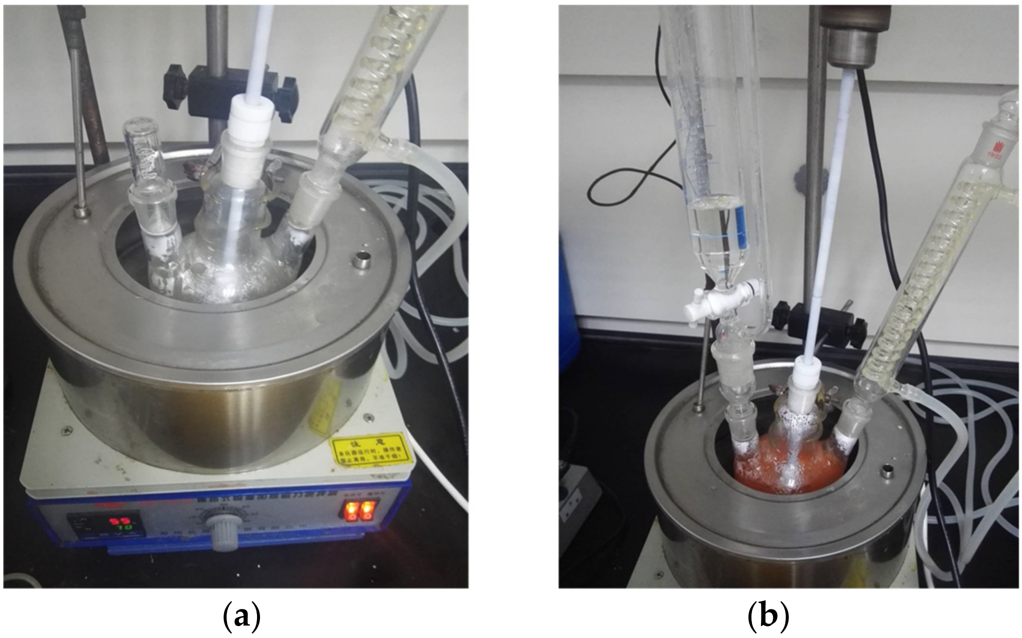
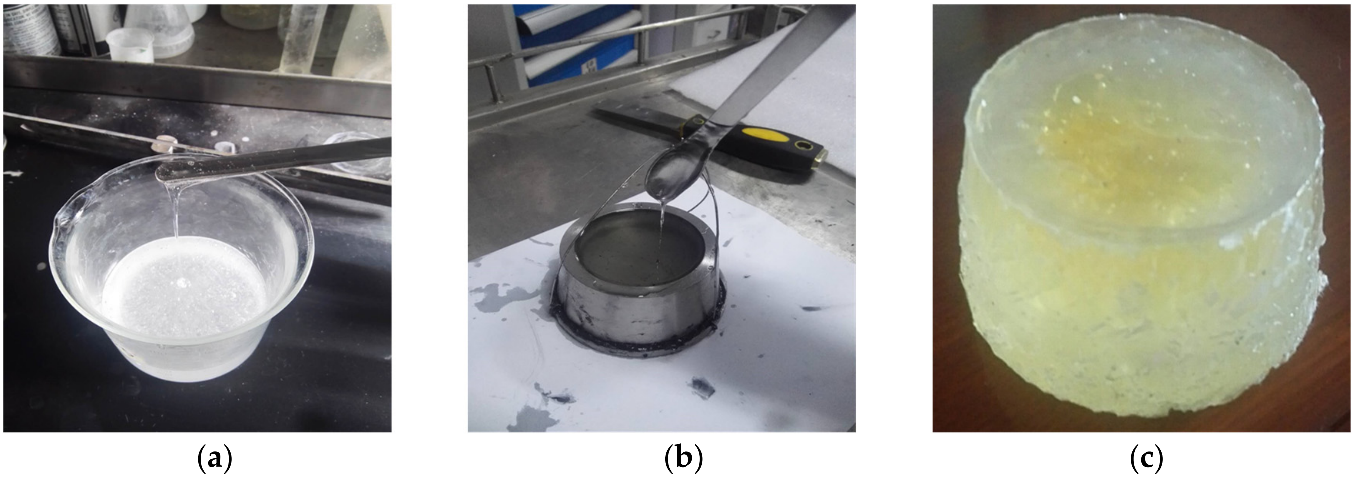

(**a**) (**b**)

Figure 1. Illustration of curing agent synthesis setup. (**a**) Synthetic reaction equipment. (**b**) Synthetic curing agent.

2.3. Water Bath Maintenance of the Sealant and Curing Time Test

The sealant curing experimental maintenance temperature was set at 20, 35, and 50 °C, considering the temperature from the ground to the bottom of the well. The maintenance was performed in a constant-temperature water bath, and the experimental procedure is shown in Figure 2. To begin, the weighed modified epoxy resin was combined with curing agent and viscosity reducer in a 500 mL beaker and thoroughly mixed, as shown in Figure 2a. The mixture was then slowly poured into an iron grinder with a diameter of 10 cm and a height of 5 cm, as shown in Figure 2b, and allowed to settle for some time to remove air bubbles. As shown in Figure 2c, the epoxy was removed for further testing after curing. A glass rod 20 cm long and 4 mm in diameter was used to continuously touch the slurry until the glass rod could not enter the slurry by gravity, which was the time of slurry condensation. The epoxy resin curing time was measured in underwater bath conditions at 20, 35, and 50 °C.



(**a**) (**b**) (**c**)

Figure 2. The experimental procedure of the sealant curing maintenance. (**a**) Formulation of epoxy-based polymers. (**b**) Curing state of the sealant. (**c**) Release of the sealant.

2.4. Evaluation of the Mechanical Properties

Mechanical properties are important factors in determining whether a material can be used to manage pressure in the annulus. The compressive strength and elongation at the epoxy resin system's break were evaluated to study the material's mechanical properties. The compressive strength test was performed under uniaxial stress conditions. The compressive strength is the maximum stress during damage generation in the cement stone stress-strain curve. By adjusting the spherical seat, the prepared sample is placed on the rigid pad in the center of the testing machine's bearing plate, and the rigid pad is evenly contacted with the upper and lower bearing plates. At a pressure range of 0.5–0.8 MPa, the specimens were loaded until they fractured, and then the value of the maximum stress was recorded. The uniaxial compressive strength can be calculated by using Equation (1):

$$R = \frac{P}{A} \tag{1}$$

where R represents the uniaxial compressive strength of the rock (MPa), P represents the damage load of the specimen (N), and A is the cross-sectional area of the specimen (mm^2).

Elongation at break is typically defined as the ratio of the sample's displacement at fracture to its initial length. The specimen's length before the tensile test is L_0, and after stretching the specimen grows ΔL, $\Delta L/L_0$ is referred to as the strain, which is the amount of elongation corresponding to the material's unit length. The equation for elongation at break is as follows:

$$\varepsilon = \frac{\Delta L}{L_0} \tag{2}$$

The fracture elongation of epoxy resin was examined using stripped samples according to the GB/T2567-2008 test standard using the CMT-4202 test equipment, as shown in Figure 3. Before the test, the fully cured specimen was completely remolded, and the edges of the sample strips were smoothed with sandpaper. Before the test, the samples' initial length L_0 was recorded. Then, the specimens were placed at the center of the test bench by a designated fixture. The total tensile length at fracture was calculated, as was the elongation ε.

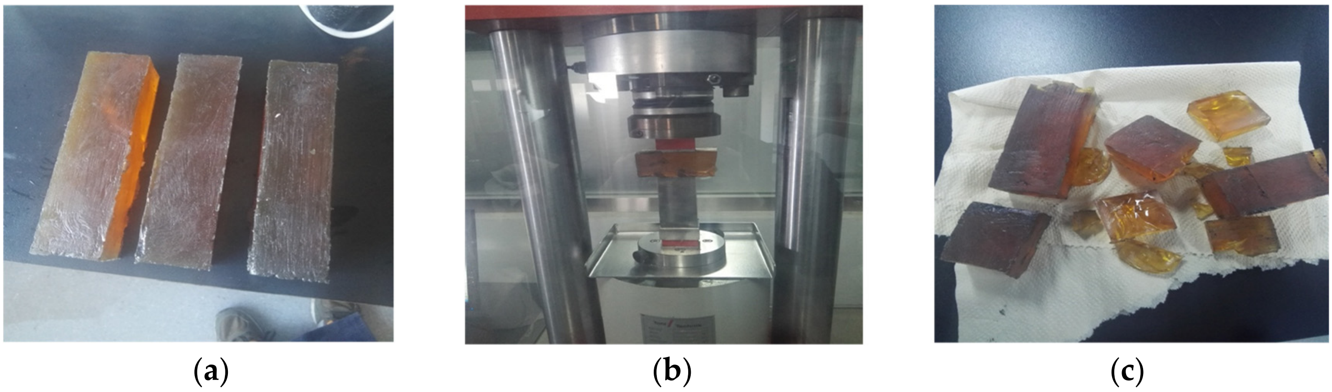

(**a**) (**b**) (**c**)

Figure 3. Sealant compressive strength tests. (**a**) Sealant specimens before the test. (**b**) Tensile test equipment. (**c**) Sealant specimens after the test.

2.5. Viscosity Evaluation

A Brinell rotational viscometer is used to measure the viscosity of the epoxy resin, as shown in Figure 4. A Brinell viscometer is comprised of an inner and outer cylinder, and the cylinder constant is marked. The liquid was poured between the two cylinders and the rotation speed under a given external torque or the external torque under a given rotation rate we measured to determine the viscosity of the liquid according to GB T 10247-2008 standard. The cylinder was chosen based on the viscosity range of the sample being measured, and the viscosity of each sample was measured three times to obtain an average value. The experimental temperature was 20 °C.

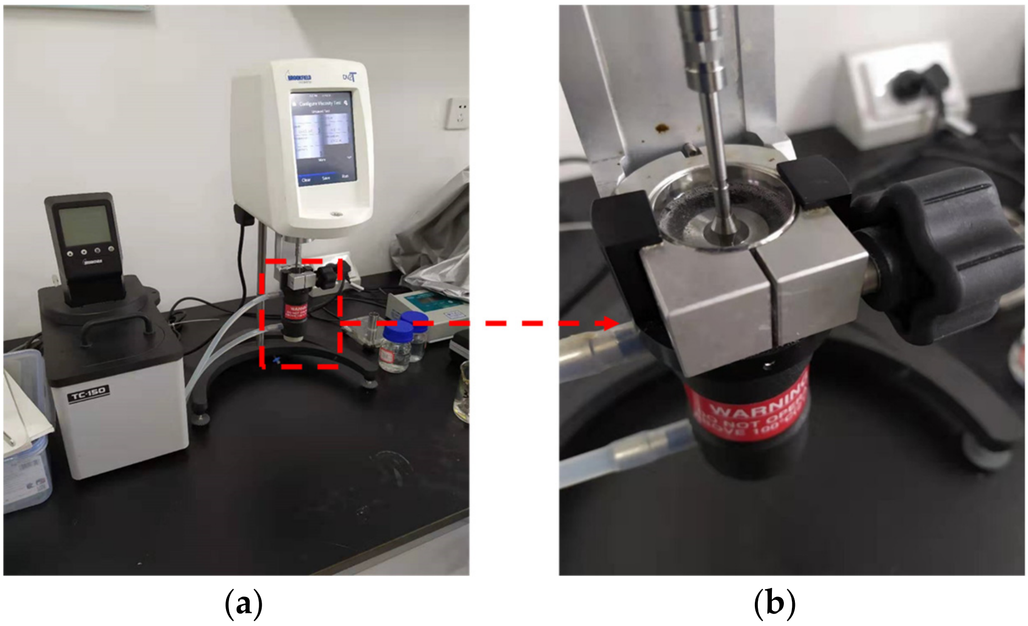

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Figure 4. Viscosity test of sealant. (**a**) Brinell rotational viscometer. (**b**) Sealant test status.

2.6. Performance Evaluation of Cement Containing Resin

The cement slurry was prepared according to the "Oil Well Cement Test Method" GB/T 19139-2012. The resin/cement composite was obtained by adding liquid epoxy resin during the slow mixing of the cement slurry. The mass ratio of resin in cement slurry was 5%, 10%, 15%, and 20%, respectively. The viscosity reducer always accounts for 25% of the resin, and two contents were added to the curing agent, which is 5% and 10%, respectively. By injecting gas into each sample, the permeability was calculated, and each sample was examined three times.

A casing annulus model was created to assess the effect of resin on conventional cement tightness. The solid cylinder and hollow casing were made of 3Cr. Figure 5 depicts the model's assembly and dimensions. The cement slurry and resin (10% curing agent) were mixed in a specific ratio and injected into the model's annulus at a height of 50 mm. After curing for 48 h, the model was transferred to the core holder. The flow chart of cement sheath sealing capability test is shown in Figure 6. Transducer 1 recorded the pressure as N_2 was continuously injected from the bottom of the holder. Transducer 2 measured the pressure in the annulus model's upper part. The experiment was ended when the pressure on both sides rose as a result of the cement sheath seal failing. The ring pressure pump pressurized the holder to ensure that it was always 2~3 MPa higher than the injection pressure, preventing gas from escaping along the inner wall of the holder during the experiment.

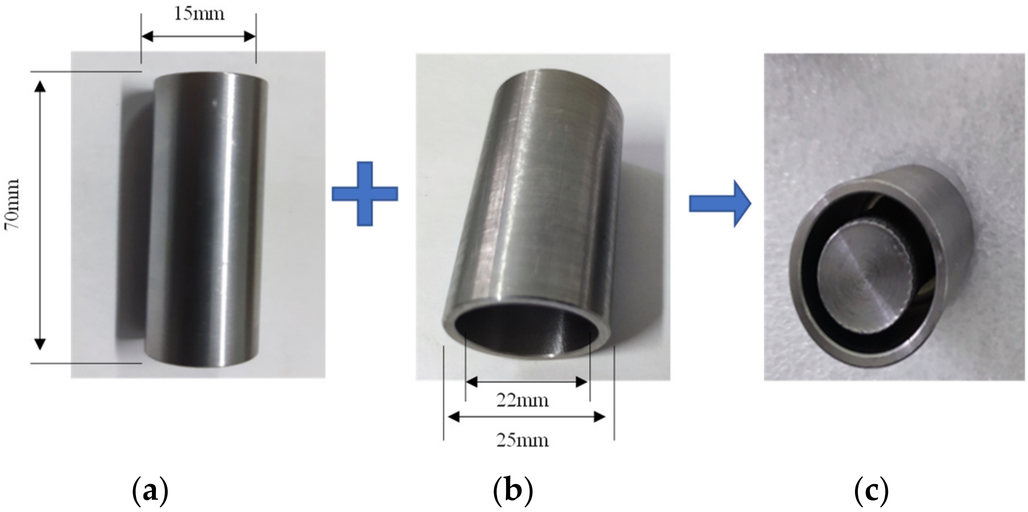


Figure 5. Assembly and dimensions of the model. (**a**) Solid cylinder. (**b**) Hollow casing. (**c**) Annular model.

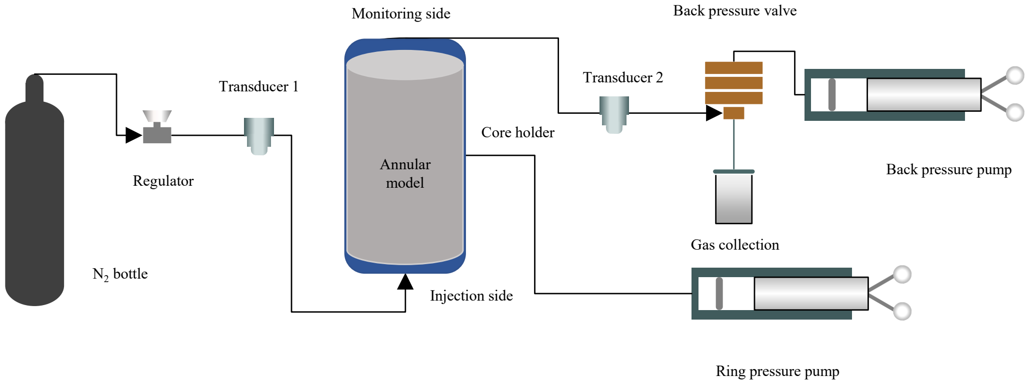


Figure 6. Test flow of cement sheath sealing ability.

3. Results

3.1. Evaluation of Viscosity Reducers

Butyl glycidyl ether (660), phenyl glycidyl ether (690), and ethylene glycol diglycidyl ether (690) were chosen as low-cost, commercially available viscosity reducers (669). Table 2 shows the results of adding 5% viscosity reducers. The results show that butyl glycidyl ether (660) has the best viscosity reduction effect on E-51 resin, and the viscosity of the resin solution after adding 5% viscosity reducer is only 1.69 Pa·s. As a result, to reduce the sealant system's viscosity as much as possible, butyl glycidyl ether (660) is used as the viscosity-reducing agent in this study.

Table 2. Viscosity of E-51 epoxy resin after adding 5% wt viscosity reducer.

System	**E51**	**E51 + 5% 660**	**E51 + 5% 690**	**E51 + 5% 669**
Viscosity (Pa·s)	35.7	1.69	2.85	3.96

The viscosity of the E-51 epoxy resin system containing different concentrations of viscosity reducer 660 concentration was studied to determine further the optimal viscosity of viscosity reducer 660 addition concentration, and the results are shown in Figure 7. It can be seen that as the viscosity reducer concentration increases, the sealant viscosity decreases rapidly. The sealant system's viscosity is 35.7 Pa·s without viscosity reducer 660. When a 25% viscosity reducer is added, the sealant system's viscosity is 0.061 Pa·s. In this study, a viscosity reducer content of 25% is used to facilitate the sealant's entry into the cement ring microcracks. The viscosity-reducing agent content of the sealant formulations is 25% unless otherwise specified below. It is important to note that the viscosity of each formulation increases slightly with the passage of time, which is caused by the volatilization of the 660 components of the viscosity reducer, so it is best to mix quickly or keep the seal when mixing.

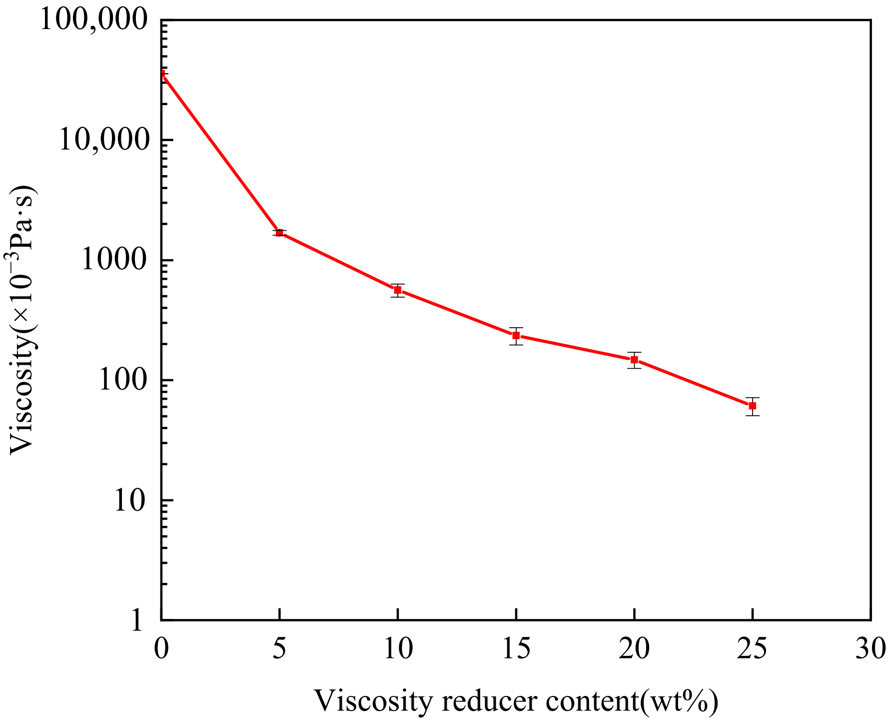


Figure 7. Sealing viscosity test curve during 660 viscosity reducer adjustment.

3.2. Density Test

The density of the sealant is an important factor in sealant performance. By varying the ratio of viscosity reducer, curing agent, and resin used to formulate the sealant, the effect of different compositions on the density of the sealant system is investigated. The experiments were conducted at room temperature (20 °C), and the results are shown in Figure 8. When the density of E-51 epoxy resin is 1.25 g/cm^3, the density of the sealant decreases as the viscosity reducer and hardener content increase. The sealant's density is reduced from 1.25 to 1.02 g/cm^3. It was discovered that the viscosity-reducing agent content has a greater influence on sealant density, owing to its lower density.

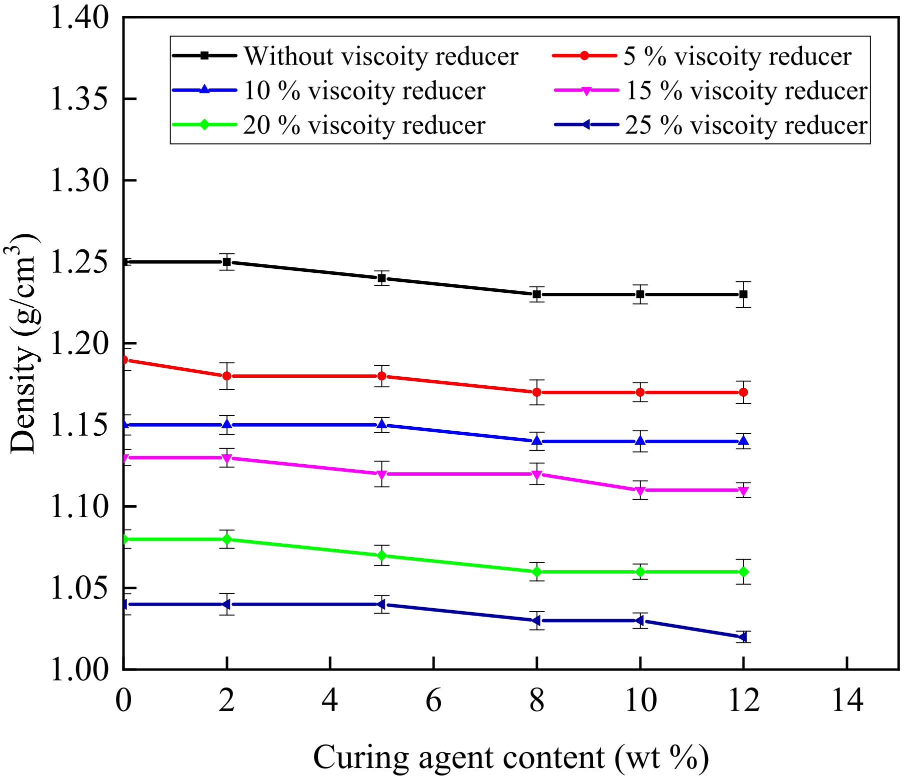


Figure 8. Density changes of the sealant system with different compositions.

3.3. Curing Time of the Sealant System

The curing time of a sealant is an important indicator of its performance, which is related to building design and safety. The curing time of sealants that were formulated by different components at different temperatures is shown in Figure 9. The higher the maintenance temperature, the higher the curing agent content, and the shorter the coagulation time. When the amine in the curing agent reacts with the epoxy group in the resin, the Arrhenius equation states that increasing the temperature and the concentration of functional groups can lower the reaction potential and accelerate the reaction rate, resulting in a shorter curing time. The curing time is controlled for 0.25–20 h by adjusting the curing agent content to 20–50 °C.

3.4. Compressive Strength and Elongation at Break of the Sealant System

Compressive strength is one of the most important and basic mechanical properties of sealants, which evaluates the resistance of the material to compression damage under high load conditions. A compressive strength test is performed under a specified temperature, humidity, and compression rate. The compressive load is applied to the specimen along the longitudinal direction to cause deformation until the material is destroyed. Determination of the yield force, breaking force, and crushing strength of the specimens is an important reference for quality control and application design. The results of the compressive strength test are shown in Figure 10. The higher the curing temperature and the higher the curing agent content, the higher the compressive strength. After 24 h curing, the compressive strength of the resins can reach 18 to 55 MPa, which is higher than ordinary resin or resin and cement mixture [21]. It can be concluded that the mechanical properties of this new sealant system are very high and adjustable.

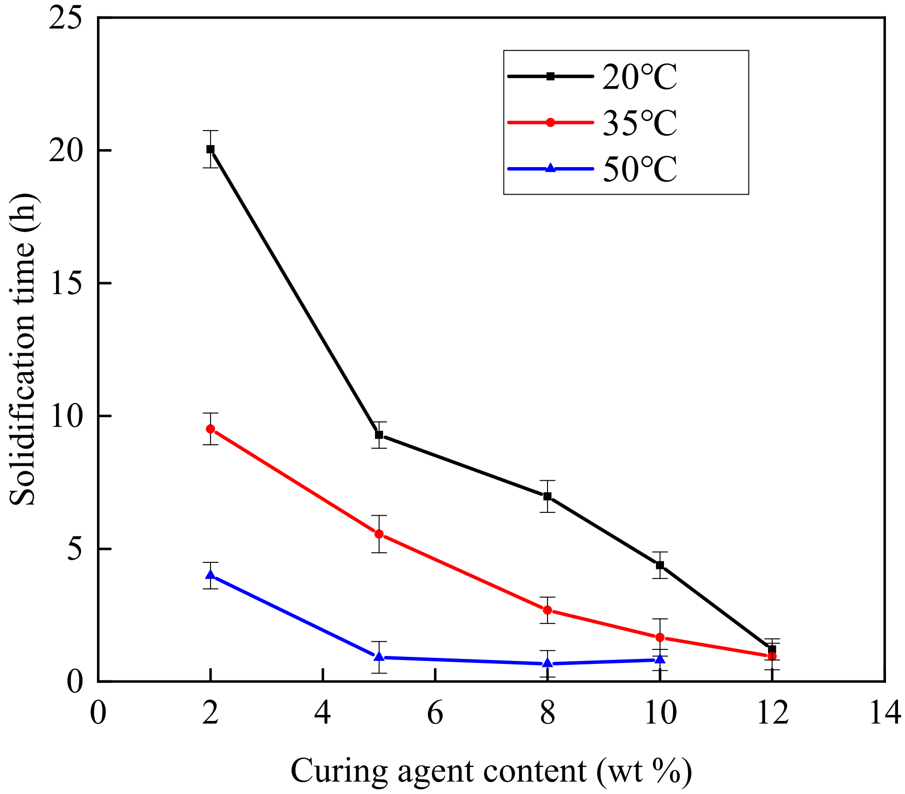


Figure 9. Curing time curve of sealant under different curing agent content.

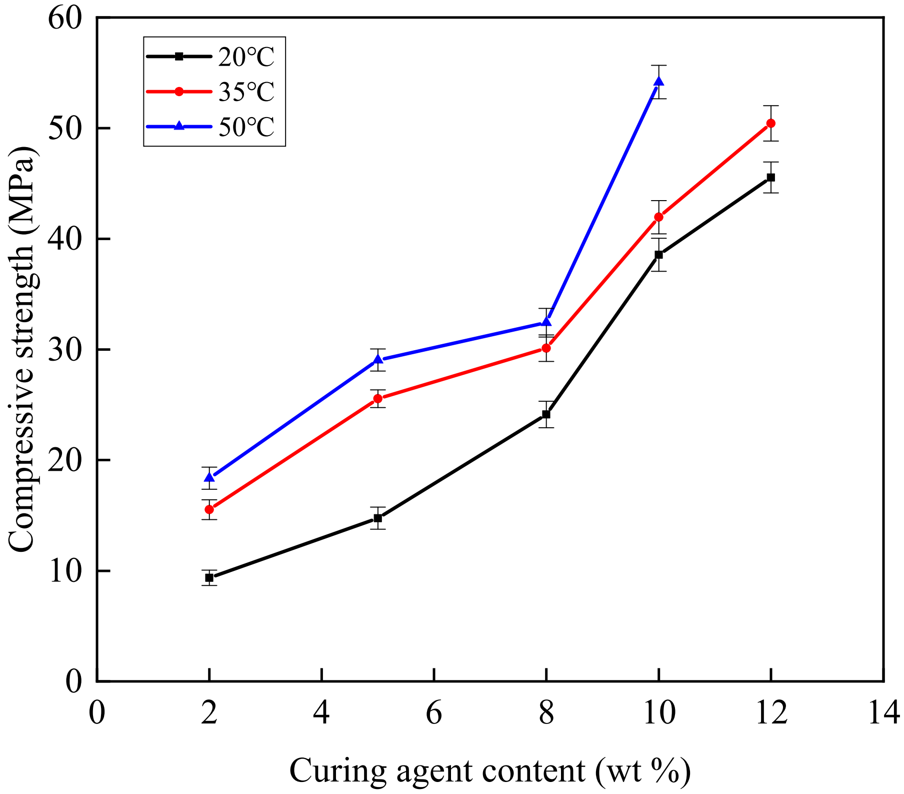


Figure 10. The compressive strength changes of sealant with varying amounts of curing agent.

The test results of elongation at break are shown in Figure 11. It can be observed that the viscosity reducer can not only lower the viscosity of the sealant but also enhance the cured product's elongation at break. The system curing product elongation at break gradually increases as the sealant viscosity decreases. When no viscosity reducer is present, the cured product elongates at a break of 1.9%. When the descending adhesive content is 25%, the elongation at the break of the cured product can reach 18%. It is clear that the sealant system has exceptional toughness.

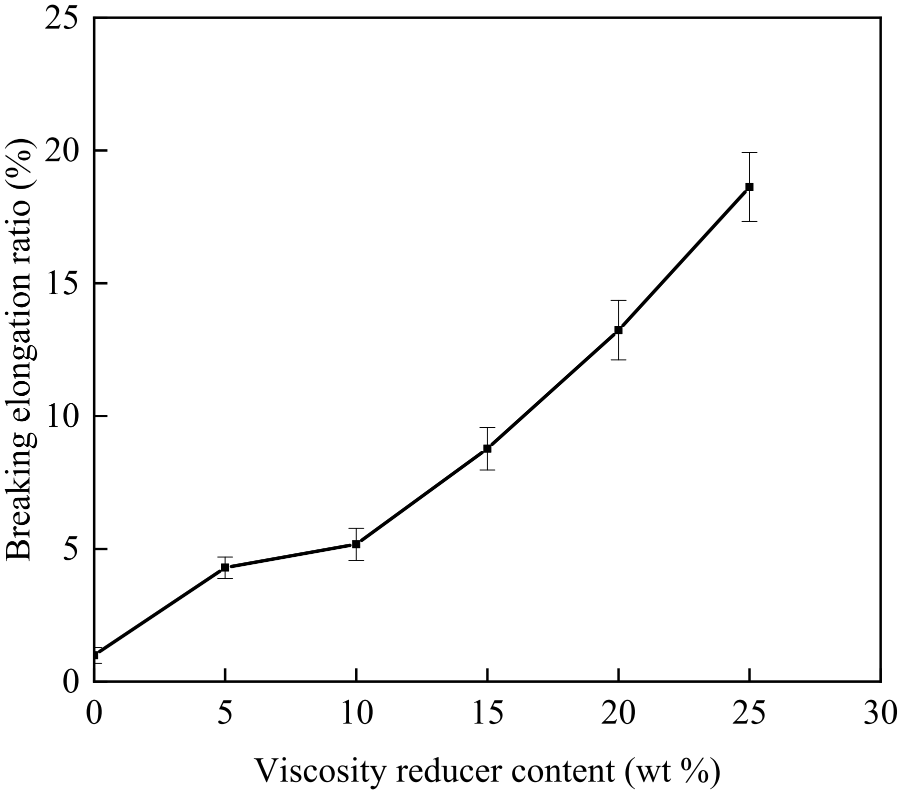


Figure 11. Change of the breaking elongation ratio of sealant.

3.5. Effect of Resin on Cement Sealing Performance

The cement permeability has a direct impact on the cement sheath's sealing ability. By lowering the cement's permeability, the difficulty of gas passing through the cement into the annulus is increased. The variation of conventional cement permeability with different resin content is shown in Figure 12. The permeability of cement is clearly reduced after adding 5% and 10% curing agents, respectively, as the resin content increases. When the resin content is 20% (10% curing agent), the permeability of cement decreases by 46.3%, from 0.041×10^{-3} μm^2 to 0.022×10^{-3} μm^2. The curing of the resin polymer occurs concurrently with cement hydration after the resin is mixed with the cement slurry. The cured resin polymer fills the cement matrix structure and forms a cross-linking structure with the hydration products of cement, thereby reducing the existence of microcracks in the cement, decreasing the permeability of cement, and enhancing the sealing ability of the cement sheath. Furthermore, when the resin content is fixed at 20% and the curing agent content is increased from 5% to 10%, the permeability of the cement decreased from 0.029×10^{-3} μm^2 to 0.022×10^{-3} μm^2, indicating that an increased curing agent content can further reduce the permeability of cement under the same resin content. This is due to the curing agent's ability to promote the polymerization reaction of the resin, resulting in a more effective filling of the cement skeleton by the resin polymer, thereby improving the sealing performance of the cement sheath and decreasing its permeability. To achieve optimal sealing performance, it is necessary to control the content of resin and curing agent in the design and preparation of cement sheaths.

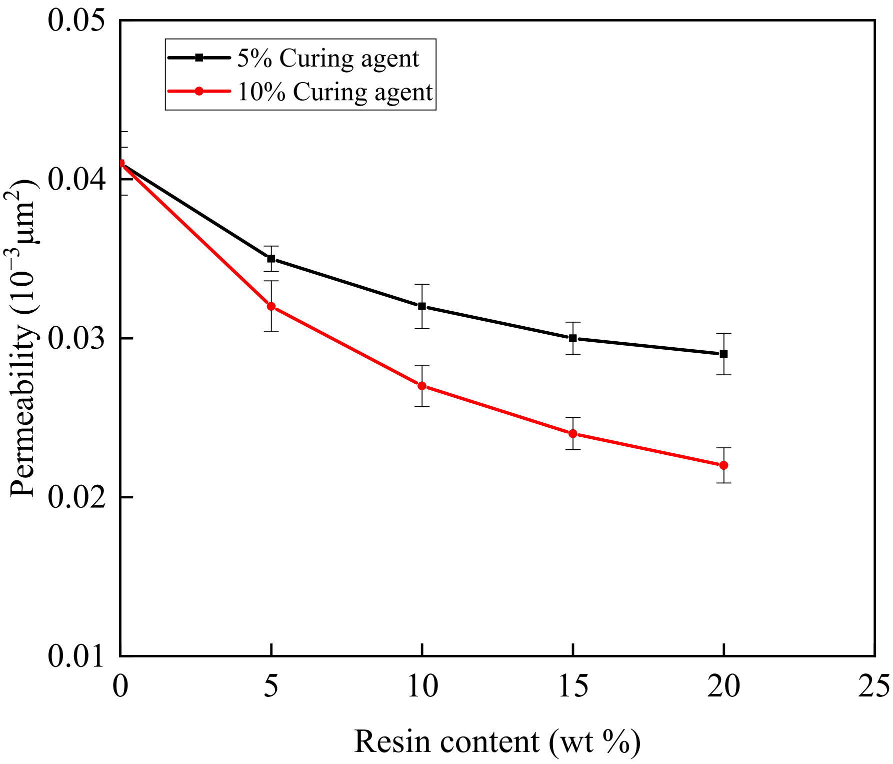
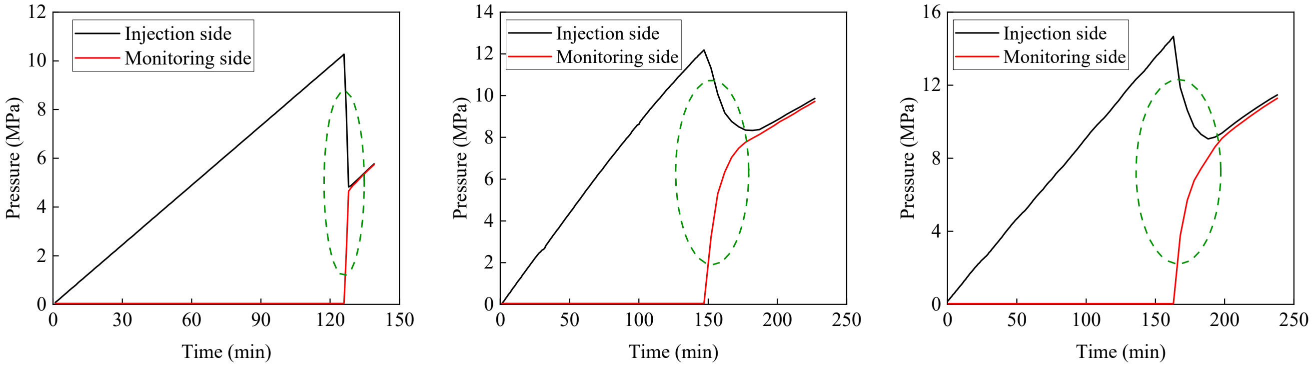


Figure 12. Permeability of conventional cement under different resin content.

A high-quality cement sheath is required for effective gas containment within the wellbore annulus. The sealing ability of the conventional cement sheath is shown in Figure 13. The injection pressure is gradually increased, and the monitoring pressure is zero before the gas passes through the cement sheath and casing combination. As the monitoring pressure rises due to gas escape, the injection pressure drops abruptly. After being basically the same, the pressure on both sides gradually increases due to the injection side's continuous gas injection. The cement sheath's maximum sealing pressure without resin is 10.3 MPa. The maximum sealing pressure of the cement sheath mixed with 10% and 20% resin is 12.2 and 14.7 MPa, respectively, which is 18.5% and 42.7% greater than the maximum sealing pressure of the pure cement sheath. It demonstrates that adding resin improves the compressive strength of the hardened cement paste and the tightness of the cement sheath. It is worth noting that the rate of change of the injection pressure and the monitoring pressure (inside the green dotted line) is clearly different after the cement sheath seal failure. Figure 13a shows two rapid pressure changes. Figure 13b,c shows relatively flat pressure changes. The occurrence of an unobstructed gas pathway through the bonding interface between the cement and casing is evidenced by the rapid convergence of the injection pressure and monitoring pressure values. Along the gas channel, the cement and casing are devoid of any interfacial bonding force. However, the addition of resin to the cement extends the time that is required for the injection pressure and monitoring pressure values to converge following the gas breakthrough, particularly when the pressure differential is small and the curve is more gradual. These observations suggest that the bonding interface retains some bonding strength even in the presence of a gas channel, resulting in a delay in the attainment of maximum monitoring pressure values and a possible reduction in the rate of pressure buildup in the wellbore annulus.



(**a**) (**b**) (**c**)

Figure 13. Sealing ability of the conventional cement sheath. (**a**) Cement sheath with 0% resin. (**b**) Cement sheath with 10% resin. (**c**) Cement sheath with 20% resin.

3.6. Applicability Analysis

As the viscosity of the epoxy resin increases and the curing reaction is more difficult to control, this brings about the problem that the application of epoxy resin cementitious materials is limited. When the resin is mixed with water, it will have a greater impact on the cross-linking reaction, releasing more heat. This can lead to local curing within minutes, triggering a burst of polymerization and creating a construction risk, as shown in Figure 14.

(**a**) (**b**)

Figure 14. (**a**) Unstable polymerization of conventional epoxy resins. (**b**) Stable curing of the modified sealants.

The curing agent that was synthesized in this study uses isophorone diamine as one of the main reagents, and the preferred viscosity reducer is a homogeneous oil-soluble butyl glycidyl ether, resulting in very little water miscibility with the resin system. The conventional resin system mixed with water exhibits a strong water solventization phenomenon and obvious miscibility. In contrast, after mixing, the modified resin system in this study forms an opaque emulsion, and phase separation will occur as time passes. Since the density of water is less than that of the resin, the water will be distributed on top of the resin, resulting in no violent exothermic reaction. The resin system is compatible with standard aqueous solutions and other liquid slurries and will not react with other systems even in brine environments.

The resin has good mechanical properties and bonding properties, and its compressive strength is greater than that of traditional cement (30~40 MPa) [31,32]. Resin is used not only as a single component but also as a resin/cement composite. Compared with conventional cement, the cement-formation interface bonding strength of the epoxy resin group increased from 0.16 MPa to 1.52 MPa [33]. Adding 12-vol% epoxy to conventional cement can improve shear bonding by 25% [20]. Therefore, the resin can act as an annular sealant to enhance the long-term integrity of the wellbore.

4. Conclusions

A solid-free resin sealant with low viscosity, excellent mechanical properties, and high chemical stability was developed in this study. In addition, a suitable viscosity reducer 660 was introduced into the resins to further improve its performance. The following conclusions can be drawn from the experimental results:

1. The viscosity of the sealant can be adjusted from 35.7 Pa·s to less than 0.065 Pa·s by adding viscosity reducer 660. The decrease in the resin viscosity increases the chance of it penetrating into microfractures in the cement ring.
2. The higher the temperature, the higher the curing agent content, and the shorter the sealant curing time. The curing time can be adjusted within 0.25–20 h by controlling the dosage of curing agents to meet different field requirements.
3. The sealant system has excellent mechanical properties. The higher the curing temperature, the higher the content of curing agents, and the higher the compressive strength. After 24 h curing, the compressive strength of the resins can reach 18 to 55 MPa, which is better than conventional cement. The permeability of cement combined with 20% resin decreases by 46.3%, while the sealing pressure increases by 42.7%. The addition of resin compacts the cement's internal structure, which can enhance the sealing performance of the wellbore annulus.

Author Contributions: Methodology, N.D.; investigation, Z.W.; resources, G.L.; data curation, H.L.; writing—original draft preparation, Z.H. and N.D.; writing—review and editing, K.J. and Z.W.; visualization, N.D. All authors have read and agreed to the published version of the manuscript.

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