

Preparation and properties of alcohol-eliminating one-component silicone sealant for interior decoration

Li Ziyong¹, Chen Bingyao^{1,2,3}, Li Yan¹, Zheng Jiadong¹, Fang Yamin¹

(1. Guangdong Sanhe Holding Co. LTD., Zhongshan 528400, Guangdong, China;

2. Guangdong Sanhe Chemical Technology Co. LTD., Foshan 528400, Guangdong, China ;

3. Sanhe Fine Chemical (Guangdong) Technology Co. LTD., Zhongshan 528400, Guangdong, China)

Abstract: Using α,ω -dihydroxy polydimethylsiloxane as the base polymer, methyltrimethoxysilane as the crosslinking agent, dimethyldimethoxysilane as the chain extender, and tetraisopropyl titanate as the catalyst, an alcoholremovable onecomponent silicone sealant for interior decoration was prepared. Singlefactor experiments were carried out to systematically investigate the effects of crosslinking agent dosage, chain extender dosage, catalyst dosage, filler ratio and coupling agent type on the curing behavior, mechanical properties and multisubstrate adhesion performance of the sealant. The results show that the optimal balance between strength and elasticity is achieved at 8 parts of crosslinking agent. The difunctional chain extender can effectively reduce crosslinking density and improve elongation at break and elastic recovery rate, while there exists a critical upper limit for its dosage. When the dosage exceeds 6 parts, the sealant fails to cure due to the inability to form a three-dimensional crosslinked network. At the optimal dosage of 4 parts, the tensile strength is 1.88 MPa, the elongation at break is 568 %, the elastic recovery rate is 92 %, and the 100 % modulus is only 0.28 MPa. Increasing the dosage of nanocalcium carbonate can significantly increase the elongation at break. The maximum tensile strength of 1.88 MPa is obtained at the filler ratio of 80:40. With the reinforcement of 10 parts fumed silica, the tensile strength is further increased to 2.13 MPa. When coupling agents KH-550 and KH-560 are compounded at a mass ratio of 70 to 30, 100 % cohesive failure is observed on four typical interior substrates including glass, ceramic tile, pine wood and aluminum alloy, demonstrating excellent universal adhesion performance. This study provides experimental basis for the formulation design of lowmodulus multisubstrate universal silicone sealant for interior decoration.

Key words: silicone sealant; interior decoration; alcohol-free; chain extender; bonding performance

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Indoor decoration involves sealing numerous joints such as those in kitchens, bathrooms, door and window frames, skirting boards, and suspended ceilings. Silicone sealant, with its excellent elasticity, water resistance, and durability, has become the preferred material. Unlike its outdoor application in building curtain walls, silicone sealant used for indoor decoration requires low odor, low volatile organic compound (VOC) emissions, environmental friendliness, and non-toxicity. It must also form reliable bonds with various common substrates such as glass, tiles, stone, wood, and aluminum alloy. Traditional deacidified and deketoxified silicone sealants

release acetic acid and butanone oxime, respectively, which have pungent odors and high corrosivity risks, limiting their use in indoor applications. Dealcoholized room temperature vulcanizing silicone sealants produce methanol or ethanol as crosslinking by-products, which are non-corrosive and have a low odor. This is an ideal technical route for indoor decoration sealants.

Currently, de-alcoholized silicone sealants still exhibit

Biography: Li Ziyong (1996–), Assistant engineer, mainly engaged in the research of silicone adhesives.

inherent deficiencies in storage stability and curing speed. Meeting the requirements of high strength, high elasticity, and low modulus poses a challenge in formulation design. Liu Congpan et al. prepared a rapid-curing de-alcoholized silicone sealant using methyltrimethoxysilane as a crosslinking agent and found that α -functional silane can achieve surface dryness within 2 minutes, with a tensile strength of up to 2.26 MPa. In terms of low modulus regulation, chain extenders, as bifunctional reactive silanes, can be embedded into the crosslinked network to increase the segment length between crosslink points, thereby improving flexibility and elastic recovery without significantly sacrificing strength. However, excessive use can disrupt the three-dimensional network structure, leading to failure to cure. Mao Yunzhong et al. introduced ZL-08 chain extender into the silicone sealant system, verifying its effect of reducing hardness and increasing elongation. Currently, systematic studies on the application of chain extender strategies to de-alcoholized silicone sealants for interior decoration, while simultaneously investigating their impact on the bonding performance of various indoor substrates, are still scarce.

In this paper, 107 silicone rubber was used as the base rubber, methyltrimethoxysilane as the crosslinking agent, dimethyldimethoxysilane as the chain extender, and tetraisopropyl titanate as the catalyst to prepare a de-alcoholized one-component silicone sealant. The formulation was optimized through single-factor experiments, with a focus on exploring the influence of the amount of chain extender on curing characteristics, mechanical properties, and general bonding performance. Finally, a general-purpose sealant with comprehensive performance meeting the needs of interior decoration was obtained.

1 Experimental part

1.1 Experimental raw materials

α,ω -dihydroxyl polydimethylsiloxane, viscosity 20000 mPa·s, Shandong Dongyue Organosilicon Materials Co., Ltd.; methyltrimethoxysilane, industrial grade; dimethyldimethoxysilane, industrial grade, Hubei Xintianlan New Materials Co., Ltd.; tetraisopropyl titanate, chemically pure, Shandong Heruidong Fine Chemicals Co., Ltd.; nano calcium carbonate (NPCC), CCS-25 type, particle size

approximately 80 nm, specific surface area approximately 20 m²/g, Guangxi Warner New Materials Co., Ltd.; heavy calcium carbonate (GCC), CC-1250 type, 1250 mesh, Guangxi Building Materials (Jiaoling) Fine Chemicals Co., Ltd.; fumed silica, specific surface area 200±25 m²/g, Wacker Chemie; dimethyl silicone oil, viscosity 350 mPa·s; γ -aminopropyl triethoxysilane, γ -(2,3-epoxypropoxy)propyl trimethoxysilane, Nanjing Liangui Chemical Co., Ltd.

1.2 Main instruments and equipment

Planetary mixer: XJB-2.5, Foshan Jin Yinhe Intelligent Equipment Co., Ltd.; Universal testing machine: WDS-20, Jinan Zhonglu Chang Testing Machine Manufacturing Co., Ltd.; Hardness tester: LX-A, Wuxi Xijing Rubber and Plastic Measuring Instrument Factory; Thick paint putty consistency tester: QCT, Tianjin Zhongya Material Testing Machine Factory.

1.3 Sample preparation

Add 100 parts by mass of 107 silicone rubber, 25 parts of dimethyl silicone oil, and metered amounts of nano-calcium carbonate, heavy calcium carbonate, and fumed silica into a planetary mixer. Stir and dehydrate for 3 hours under conditions of vacuum degree of -0.09 MPa and temperature above 110°C. After dehydration, stop heating and continue to cool to room temperature under vacuum stirring. Then add a metered amount of chain extender, dimethyldimethoxysilane, and stir in vacuum for 10 minutes to ensure uniform dispersion. Subsequently, add the crosslinking agent methyltrimethoxysilane and coupling agent in sequence, and stir in vacuum for 10 minutes. Finally, add the catalyst tetraisopropyl titanate, stir in vacuum for 5 minutes, defoam, and transfer to a sealed polyethylene plastic cylinder for storage.

1.4 Testing and characterization

The surface dry time is tested according to GB/T 13477.5—2002; the curing depth is tested according to GB/T 13477.6—2002; the tensile strength and elongation at break are tested according to GB/T 528—2009, using Type II dumbbell specimens, with a tensile rate of 500 mm/min; the Shore A hardness is tested according to GB/T 531.1—2008; the 100% modulus at a fixed extension is read from the tensile curve; the tensile bond strength is tested according to GB/T 13477.8—2002, with substrates of glass, ceramic tile, pine wood, and aluminum alloy, respectively, and the failure mode

is recorded; the elastic recovery rate is tested according to GB/T 13477.17—2002, with a tensile width of 100%; the VOC content is determined by referring to Appendix F of GB 18583—2008; thermal aging performance: the change rate of tensile properties is tested after aging at 80 °C for 168 hours.

2 Results and discussion

2.1 The influence of crosslinking agent dosage on mechanical properties

The crosslinking agent, methyltrimethoxysilane, is a trifunctional silane, and its dosage directly determines the crosslinking density. Table 1 lists the tensile property data of sealant with a crosslinking agent dosage ranging from 6 to 12 parts.

Table 1 Effect of crosslinking agent dosage on the tensile properties of sealant

Crosslinking agent/portion	Tensile strength / MPa	Elongation at break, %	100% modulus / MPa	Shore A hardness
6	1.42	568	0.28	16
8	1.76	485	0.35	21
10	1.83	380	0.46	27
12	1.90	290	0.58	33

2.2 The influence of chain extender dosage on curing characteristics and cross-linking network

8 parts of a fixed crosslinking agent are used, and a bifunctional chain extender, dimethyldimethoxysilane, is introduced. The two ends of the chain extender contain hydrolyzable methoxy groups, which can condense with the hydroxyl groups of the base rubber or the hydrolysis products of the crosslinking agent, thereby extending the chain segments and reducing the crosslinking density. However, when the amount of chain extender is excessive, it competes with the trifunctional crosslinking agent for the consumption of active hydroxyl groups and hydrolysis sites, resulting in insufficient three-dimensional crosslinking points and an inability for the network to penetrate. The cured state and mechanical properties under different amounts of chain extender are shown in Table 2.

When the amount of chain extender is increased from 0 to 4 parts, the tensile strength increases from 1.76 MPa to 1.88 MPa, the elongation at break increases significantly from 485% to 568%, and the elastic recovery rate increases from 87% to 92%. The increase in the segment length between

As the amount of crosslinking agent increases, the tensile strength rises from 1.42 MPa to 1.90 MPa, with a monotonic increase in 100% modulus and hardness, while the elongation at break decreases significantly from 568% to 290%. The increase in crosslinking density restricts the movement of molecular chains, enhancing the rigidity of the network. When the amount is 8 parts, the tensile strength is 1.76 MPa, the elongation at break is 485%, the 100% modulus is 0.35 MPa, and the mechanical balance is optimal. In the dealcoholized system, there is an optimal amount of crosslinking agent, where too little results in insufficient strength and too much results in severe sacrifice of elongation at break. This paper chooses an amount of 8 parts of crosslinking agent as the basis for subsequent research.

crosslinking points makes the network more flexible and the network structure more complete, ensuring uniform stress transfer and achieving both toughening and reinforcement. When the amount of chain extender is 6 parts, the tensile strength decreases to 1.67 MPa, but an elastomer can still be formed. When the amount reaches 8 parts, the system cannot be fully cured, and the subsurface layer exhibits a viscous flow state. The reason is that excessive bifunctional active groups compete with the crosslinking agent, and the concentration of crosslinking points is below the gelation threshold, causing the system to lose the ability to form a three-dimensional network and only producing linear or branched oligomers. Therefore, the safe upper limit of dimethyldimethoxysilane (DMS) in this system is 6 parts, and the optimal amount for comprehensive performance is determined to be 4 parts.

2.3 Influence of catalyst dosage

Tetraisopropyl titanate is a commonly used catalyst for dealcoholized RTV silicone sealant, and its dosage significantly affects the curing rate and storage stability. The results are shown in Table 3.

When the catalyst dosage is 1.5 parts, the surface drying time is 22 minutes, and the curing depth after 7 days is 2.9 mm.

Table 2 The influence of chain extender dosage on the curing state and mechanical properties of sealant

Chain extender/ portion	Cured state	Surface drying time/min	Curing depth (in mm)	Tensile strength/ MPa	Elongation at break, %	100% modulus / MPa	Elastic recovery rate/%
0	Fully cured	15	3.8	1.76	485	0.35	87
2	Fully cured	18	3.4	1.82	520	0.32	89
4	Fully cured	22	2.9	1.88	568	0.28	92
6	Fully cured	28	2.3	1.67	598	0.24	90
8	Surface solidification, internal viscous flow	40	0.5	—	—	—	—

Note: After being cured for 14 days, the surface of the 8 samples was solidified, but the deep layer remained in a viscous flow state, making it impossible to cut into shapes for mechanical testing.

The storage is normal, and the balance between curing rate and storage stability is good. Titanate is extremely sensitive to moisture. Too high a dosage can catalyze the slow condensation of polymers in sealed storage, leading to a shortened storage period. Therefore, the catalyst dosage is determined to be 1.5 parts.

2.4 Regulation of mechanical properties by filler system

Based on 8 parts of crosslinking agent, 4 parts of chain extender, and 1.5 parts of catalyst, the ratio of nano-calcium carbonate (NPCC) to ground calcium carbonate (GCC) was first investigated. The results are shown in Table 4.

Table 4 The influence of filler ratio on mechanical properties

NPCC:GCC	Tensile strength / MPa	Elongation at break/%	100% modulus /MPa
60:40	1.42	485	0.35
80:40	1.88	568	0.28
100:40	1.75	625	0.24

As can be seen from Table 4, when the amount of nano-calcium carbonate increases from 60 parts to 100 parts, the elongation at break continuously increases from 485% to 625%, while the modulus at 100% remains unchanged from 0.35 MPa to 0.24 MPa. With a particle size of approximately 80 nm, nano-calcium carbonate undergoes surface treatment with organic modifiers such as stearic acid. The organic

Table 3 Effect of catalyst dosage on curing characteristics and storage stability

Catalyst/portion	Surface drying time/min	7 d curing depth/mm	State after storage at 50°C for 7 days
1.0	35	2.0	Normal, can be extruded
1.5	22	2.9	Normal, can be squeezed out
2.0	15	3.3	Slight thickening
2.5	10	3.6	Significant thickening and difficulty in extrusion

segments on its surface can physically entangle and adsorb with the molecular chains of silicone polymers, forming reversible physical cross-linking points. During stretching, these physical cross-linking points can undergo slippage and reconstruction, effectively dissipating strain energy and delaying crack propagation, thereby significantly improving the elongation at break. The tensile strength reaches a maximum of 1.88 MPa when the ratio is 80:40, and decreases to 1.75 MPa when the amount of nano-calcium carbonate is further increased to 100 parts. This is because an appropriate amount of nano-calcium carbonate can form a uniform filler network, effectively transmit and disperse stress, and thus enhance the material; however, when the content of nano-filler is too high, the distance between particles decreases, and the van der Waals force increases, leading to easy agglomeration and formation

of stress concentration points, which in turn results in a decrease in tensile strength. Considering the balance between tensile strength, elongation at break, and modulus, the optimal ratio of nano-calcium carbonate to heavy calcium carbonate is determined to be 80:40.

2.5 The influence of coupling agent types on the bonding performance of multiple substrates

Indoor decoration joints involve various substrates such as glass, ceramic tiles, wood, and aluminum alloy, requiring sealants with universal good adhesion. Table 5 presents the tensile bond strength and failure mode of sealants on various substrates under different coupling agent systems.

As can be seen from Table 5, KH-550 exhibits the most significant improvement in bonding to polar substrates such

as wood and ceramic tiles, due to its primary amino groups capable of forming hydrogen bonds or covalent bonds with the hydroxyl groups on the substrate surface. KH-560 is beneficial for improving interfacial wetting and enhancing water-resistant bonding, but its reactivity alone is insufficient. When mixed in

a 70:30 ratio, the combination achieves 100% cohesive failure for all substrates, with the highest bonding strength, exhibiting true "universal" bonding characteristics. Therefore, the coupling agent system is set to KH-550/KH-560=70:30, with a dosage of 1.2 parts.

Table 5 Influence of coupling agent types on adhesion performance of multiple substrates

Coupling agent system	Glass substrate		Ceramic tile substrate		Pine substrate		Aluminum alloy substrate	
	Strength / MPa	Destruction form	Strength / MPa	Destruction form	Strength / MPa	Destruction form	Strength / MPa	Destruction form
No coupling agent	0.82	Interface damage	0.68	Interface failure	0.55	Interface failure	0.49	Interface failure
KH-550	1.35	100% cohesive	1.28	100% cohesive	1.1	90% cohesion	1.2	100% cohesive
KH-560	1.18	80% cohesion	1.05	Mixed failure	0.92	Mixed failure	0.95	Mixed failure
KH-550/KH-560 70/30	1.42	100% cohesive	1.38	100% cohesive	1.22	100% cohesive	1.32	100% cohesive
KH-550/KH-560 50/50	1.35	100% cohesive	1.25	100% cohesive	1.05	80% cohesion	1.18	100% cohesive
KH-550/KH-560 30/70	1.12	90% cohesion	1.02	Mixed failure	0.85	Interface damage	0.92	Mixed failure

3 Conclusion

This article prepared a one-component, alcohol-dehydrated silicone sealant for indoor decoration, using 107 silicone rubber as the base rubber, methyltrimethoxysilane as the crosslinking agent, and dimethyldimethoxysilane as the chain extender. When the amount of crosslinking agent was 8 parts, the sealant achieved an optimal balance between strength and elasticity. The bifunctional chain extender could effectively reduce the crosslinking density, improve the elongation at break and elastic recovery rate, but there was a critical upper limit to its amount. When the amount exceeded 6 parts, the sealant could not cure due to the inability to form a three-dimensional crosslinking network; the optimal amount was 4 parts, with a tensile strength of 1.88 MPa, an elongation at break of 568%, an elastic recovery rate of 92%, and a 100%

modulus at a fixed extension of only 0.28 MPa, achieving a synergistic optimization of high strength and high elasticity under low modulus. After surface organic modification of nano-calcium carbonate, its surface organic segments could form reversible physical crosslinking points with polymer chains, dissipating energy through slipping and reconstruction during stretching. Therefore, increasing its amount could significantly improve the elongation at break; when the ratio of nano-calcium carbonate to heavy calcium carbonate was 80:40, the tensile strength reached its maximum. When coupling agents KH-550 and KH-560 were mixed in a ratio of 70:30, the sealant achieved 100% cohesive failure on four substrates: glass, ceramic tile, pine wood, and aluminum alloy, exhibiting excellent universal bonding properties.