The influence of silane coupling agents on the properties of silicone sealants

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Abstract: Silicone sealant was prepared using α,ω-dihydroxyl polydimethylsiloxane, dimethyl silicone oil, nano-calcium carbonate, methyltrimethoxysilane, silane coupling agent, and catalyst as raw materials. The effects of four silane coupling agents, namely γ-aminopropyl triethoxysilane (KH550), γ-(2,3-epoxypropoxy)propyl trimethoxysilane (KH560), N-β-(aminoethyl)-γ-aminopropyl trimethoxysilane (KH792), and γ-mercaptopropyl trimethoxysilane (KH590), on the surface dry time, mechanical properties, and adhesion performance to substrates such as PP, PE, and PET of the silicone sealant were investigated. The results showed that the addition of KH550 and KH792 could shorten the surface dry time of the sealant and accelerate the curing speed within 24 hours, but the improvement in mechanical properties was limited, and the adhesion effect to substrates was poor. The addition of KH560 and KH590 could effectively enhance the adhesion effect to substrates, but the mechanical properties of the sealant with KH590 were slightly inferior to those with KH560. The silicone sealant with KH560 exhibited the best comprehensive performance.

Key words: silane coupling agent; sealant; bonding performance; silicone

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0 Preface

Silicone sealant is a crosslinkable elastomer produced through condensation reaction under specific conditions, using α,ω-dihydroxyl polydimethylsiloxane (107 silicone rubber), reinforcing fillers, crosslinking agents, silane coupling agents, and catalysts as raw materials. The molecular structure of silane coupling agents contains two groups with distinct chemical properties. One is the inorganic-philic group, capable of reacting with inorganic substances; the other is the organic-philic group, which can not only react with polysiloxane or other polymers but also achieve mutual dissolution through hydrogen bonding. Due to this unique structural characteristic, silane coupling agents can effectively improve the interfacial interaction between inorganic and organic molecules, significantly enhancing the composite properties of materials. As a result, silane coupling agents can enhance the bonding performance of silicone sealant to different substrates, expanding its application fields beyond the construction industry to automotive, household appliances, electronics, photovoltaics, medical devices, aerospace, and other fields. This article mainly explores the effects of different types of silane coupling agents on the surface drying time, 24-hour curing speed, mechanical properties, and bonding performance of silicone sealant to substrates such as PVC, PP, PE, PET, PMMA, ABS, and anodized aluminum.

1 Experimental part

1.1 Test raw materials

α,ω-Dihydroxyl polydimethylsiloxane: viscosity (25℃) 20,000~80,000 MPa·s, BlueStar Chemical New Materials

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Jiangxi Xinghuo Silicone Plant; dimethyl silicone oil, viscosity (25℃) 350 MPa·s, Jinan Chuangshi Chemical Co., Ltd.; nano-calcium carbonate: average particle size 60~100nm, Zhejiang Huixiang New Material Technology Co., Ltd.; methyltrimethoxysilane, industrial grade, Hubei Kewode Chemical Co., Ltd.; organic titanium catalyst, industrial grade, Shanghai Maireal Chemical Technology Co., Ltd.; γ-aminopropyl triethoxysilane (KH550), γ-(2,3-epoxypropoxy) propyltrimethoxysilane (KH560), N-β-(aminoethyl)-γ-aminopropyltrimethoxysilane (KH792), industrial grade, Hubei Xintianlan New Material Co., Ltd.; γ-mercaptopropyltrimetho xysilane (KH590), industrial grade, Nanjing Liangui Chemical Co., Ltd.

1.2 Test instruments

Laboratory planetary dynamic mixer, Foshan Sujia Yi Intelligent Equipment Co., Ltd.; Laboratory small vacuum kneading machine, Foshan Nanhai Jinchangtai Machinery Equipment Co., Ltd.; Electrically heated constant temperature blast drying oven, Shanghai Shanzhi Instrument Equipment Co., Ltd.; LK-103B electronic universal mechanical testing machine, Dongguan Likong Instrument Technology Co., Ltd.

1.3 Experimental preparation

1.3.1 Preparation of silicone sealant base material

Firstly, α,ω-dihydroxyl polydimethylsiloxane and dimethyl silicone oil are put into a vacuum kneader together, and the high-speed stirring program is initiated. After the materials are fully mixed evenly, nano-calcium carbonate is added, and high-speed stirring is continuously maintained. As the stirring progresses, when the material temperature gradually rises to 120 ℃, the vacuum function is turned on, and high-speed stirring continues for 3 hours under vacuum. After going through the above steps, the stirring is completed, and the base material preparation is finished.

1.3.2 Preparation of silicone sealant

After the base material has cooled to room temperature, it is transferred to the mixing cylinder of the planetary power mixer and stirred for 5 minutes. Subsequently, the crosslinking agent is added, and the equipment is evacuated to -0.90 MPa. Stirring continues for another 15 minutes. After thorough mixing, additives such as coupling agents and catalysts are added, and the equipment is evacuated again to -0.90 MPa. Stirring is maintained for 20 minutes to ensure uniform dispersion of all components. Finally, the mixed glue sample is filled into plastic glue bottles to complete the preparation process.

1.4 Performance testing and characterization

1.4.1 Surface drying time

The test shall be conducted according to Method B specified in the GB/T 13477.5—2017 standard.

1.4.2 Degree of curing

The test shall be conducted according to Method 3 (Curing Cup Method) specified in the GB/T 32369-2015 standard.

1.4.3 Storage stability

According to the GB/T 3512-2001 standard, the samples were subjected to thermal aging treatment at 80°C for 72 hours in an electrothermic constant temperature forced-air drying oven. Their storage stability was assessed by comparing the surface drying time and the degree of curing after 24 hours.

1.4.4 Tensile strength, elongation at break

According to the GB/T 528-2009 standard, the test sample is cut into a dumbbell-shaped Type 1 specimen, and is measured using a tensile testing machine that complies with ISO 5893.

1.4.5 Shore A hardness

According to the GB/T 531-1999 standard, the test sample should be made with a thickness greater than 6 mm, cured for 168 hours, and then undergo hardness testing.

1.4.6 Adhesion of substrate 1

The measurement shall be conducted according to the mold-free method described in 5.2.1 of the GB/T 43747-2024 standard, "Evaluation of Sealant Adhesion - Bead Peeling Method"

2 Results and discussion

2.1 The influence of silane coupling agent on the surface drying time of silicone sealant

According to the data in Table 1, the use of different types of silane coupling agents has a certain degree of influence on the surface drying time and curing degree of sealant. For example, the sealants without coupling agent and with KH560 and KH590 have similar surface drying times under standard conditions, but their curing speeds are different. After thermal aging, the surface drying time and curing degree of the sealant with KH590 are both increased. This is because the mercapto groups contained in KH590 can promote the siloxane condensation reaction, thereby shortening the curing time of the sealant. Compared to the sealants without coupling agent, with KH550, and with KH792, the surface drying time shows a trend of shortening, and the curing degree increases. After thermal aging, it also shows an increasing trend similar to that under standard conditions. This is because both KH550 and KH792 contain amino groups in their molecular structures. Amino groups have water-absorbing properties and can adsorb moisture from the air, accelerating the reaction and curing speed of the sealant. The hydrolytic activity of amino groups is high, and during thermal aging, the coupling agents containing amino groups undergo hydrolysis preferentially. The generated by-products, small molecules, attack the main chain of organosiloxane, leading to an extension of its surface drying time.

Table 1 Effect of silane coupling agents on the surface drying time and curing degree of sealant

Test project	Standard environment		Heat aging (80°C×72h)	
	Surface drying time/min	24-hour curing degree/mm	Surface drying time/min	24-hour curing degree/mm
Not added	15	2.83	16	2.81
KH550	12	3.27	20	3.44
KH560	7	2.95	11	3.15
KH590	8	3.02	13	3.22
KH792	8	3.47	16	3.60

2.2 The influence of silane coupling agents on the mechanical properties of silicone sealants

According to Table 2, the addition of silane coupling agents has led to varying degrees of improvement in the tensile strength and elongation at break of silicone sealants. When no silane coupling agent is added, the tensile strength and elongation at break are the lowest among the five types of sealants. After adding different types of silane coupling agents, both tensile strength and elongation at break have been enhanced to varying extents compared to the case without the addition of silane coupling agents. The reason for this is that silane coupling agents can form stable Si-O-Ca bonds with nano-calcium carbonate, which has a certain impact on the tensile strength and elongation at break of the sealant. Experiments have shown that, compared to other types of silane coupling agents, sealants with KH560 exhibit superior performance in terms of tensile strength and elongation at break. This result fully demonstrates that KH560 is the most effective in promoting the reinforcing effect of nano-calcium carbonate on silicone sealants. Furthermore, Shore A hardness mainly depends on key factors such as the amount of filler, the amount of dimethyl silicone oil, and the crosslinking density of the sealant. As can be seen from Table 2, there is little difference in Shore A hardness between sealants with and without the addition of coupling agents. Since the variable factor in this experiment is the silane coupling agent, and all other raw materials remain consistent, the experimental results are consistent with theory.

Table 2 Effect of silane coupling agents on the mechanical properties of silicone sealants

Test Project	Not added	KH550	KH560	KH590	KH792
Tensile strength/MPa	2.23	2.35	2.52	2.37	2.40
Fracture elongation/%	309	359	463	409	418
Shore A hardness	42	44	45	46	43

2.3 The influence of silane coupling agents on the adhesion of silicone sealant to substrates

According to Table 3, the sealant without silane coupling agent exhibits poor adhesion to four plastic substrates: PVC, ABS, PP, and PE, but shows good adhesion to PMMA, PET, and anodized aluminum. The main chain of silicone sealant consists of siloxane segments, which belong to a non-polar structure. The siloxane segments contain siloxane bonds that can crosslink with polar materials such as PMMA, PET, PVC, and ABS. However, the surface energy of PVC and ABS is lower than that of the sealant, making it difficult for the sealant to fully wet the substrate surface. The small contact area between the sealant and the substrate interface results in poor adhesion. PP and PE are non-polar materials with low surface energy and no active groups in their molecular chains, making it difficult for them to react with the silanol groups in the sealant. The addition of silane coupling agent to the sealant significantly improves its adhesion to substrates. Silane coupling agents contain both inorganic and organic affinity groups, serving as a "bridge" between organic sealants and inorganic substrates, thereby enhancing the adhesion of the sealant to substrates. However, the adhesion of the sealant to PP and PE substrates remains poor, possibly due to the presence of release agents on the surfaces of these substrates, which negatively affects adhesion. During testing, no primer was applied to PP and PE substrates.

Table 3 Effect of silane coupling agents on the adhesion of organic sealant substrates

Test Project	Not added	KH550	KH560	KH590	KH792
PMMA	△	△	√	△	△
PET	△	△	√	○	√
PVC	×	○	△	√	√
ABS	×	√	√	√	△
PP	×	×	×	×	×
PE	×	×	×	×	×
Anodized aluminum	○	○	△	√	○

Note: "√" indicates that after the sealant is peeled off, there is still 70% to 100% residual sealant on the substrate surface; " △ " indicates that after the sealant is peeled off, there is still 40% to 70% residual sealant on the substrate surface; "○" indicates that after the sealant is peeled off, there is still 1% to 40% residual sealant on the substrate surface; "×" indicates that after the sealant is peeled off, there is no residual sealant on the substrate surface.

3 Conclusion

The four different types of silane coupling agents, KH550, KH560, KH590, and KH792, have varying effects on the performance of silicone sealant. Compared to sealants without coupling agents and those with KH560 and KH590, KH550 and KH792, which contain amino groups, significantly accelerate the surface drying time and 24-hour curing speed of the sealant, improve tensile strength and elongation at break, but exhibit poor bonding performance to anodized aluminum. Sealants with KH560 and KH590 show little difference in surface drying time and 24-hour curing speed, as well as bonding performance to substrates, but the former slightly outperforms the latter in terms of tensile strength and elongation at break. In summary, silicone sealant with KH560 exhibits the best comprehensive performance.