Optimization of tensile strength and extrudability of silicone rubber using orthogonal experimental method

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Abstract: This paper uses the orthogonal experimental method to study the effects of three factors (107 glue, nano-calcium carbonate, silicone oil) on tensile strength and extrudability. Experimental results show the optimal formulation: 40% 107 glue, 60% nano-calcium carbonate, and 3% silicone oil. Under this formulation, tensile strength reaches 2.63 MPa and extrudability 68.2 mL/min. Analysis shows that silicone oil content has the greatest impact on tensile strength (40.16%), while 107 glue content has the largest effect on extrudability (64.29%). These results provide valuable guidance for silicone rubber industrial production.

Key words: orthogonal experiment; silicone rubber; tensile strength; extrudability

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Orthogonal experimentation is a kind of efficient method based on orthogonal arrays for scientifically arranging multi-factor and multi-level experiments. Its core advantage lies in its ability to replace the vast and complex testing workload in comprehensive experiments with a small number of highly representative experimental combinations, thereby efficiently revealing the primary and secondary order of different influencing factors as well as the optimal product formulation or process parameter combination. In the field of research, development, and production of silicone adhesives, the application of orthogonal experimentation is extremely extensive and profound. The formulation of silicone adhesives is a typical multi-factor system, and its final performance is jointly determined by the types, dosages, and mixing process parameters of various raw materials such as base polymers (e.g., 107 silicone rubber), fillers (e.g., fumed silica), crosslinking agents, catalysts, plasticizers (e.g., silicone oil), and coupling agents.

Silicone rubber is widely used in various applications due to its excellent properties such as resistance to high and low temperatures, electrical insulation, weather resistance, non-toxicity, and physiological inertness. These include buttons and seals in the electronics and electrical industry, gaskets and hoses in the automotive sector, catheters and implants in the medical field, food processing utensils, building sealants, aerospace sealing materials, household kitchenware, as well as encapsulation and protection of new energy battery modules and photovoltaic components.

The performance focus of silicone rubber varies across different fields, with tensile strength and extrudability being the core performance indicators for industrial applications. Tensile strength is a key indicator for measuring the resistance of silicone rubber products to tensile failure, directly determining the durability, reliability, and service life of the products. A silicone rubber seal ring or gasket with excellent tensile strength can maintain structural integrity when subjected to mechanical stress, thermal expansion and contraction, or external impacts, without easily tearing or undergoing permanent deformation, thus ensuring long-term effectiveness

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Biography: Luo Weiying (2003-), female, holds a bachelor's degree and is currently primarily engaged in research and development of silicone products.

of its sealing effect or structural support function. Meanwhile, extrudability directly affects production efficiency and the appearance quality of the final product. Good extrudability means that the rubber compound is soft and moderate, with smooth and uniform extrusion, without issues such as flow interruption, unstable extrusion, or rough surface, which enables efficient manual operation or large-scale automated production. This article improves the extrudability and tensile strength of silicone rubber by adjusting the content of 107 silicone rubberin the base material, the amount of nano-calcium powder filling, and the addition of silicone oil.

Traditional single-factor experiments require extensive testing and are challenging in balancing the relationship between extrudability and tensile strength. This article addresses the difficult problem of synergistic optimization of tensile strength and extrudability in silicone rubber, and designs an L9(3^3) orthogonal experiment. The experiment sets 107 silicone rubber, nano-calcium powder filling amount, and silicone oil addition as three factors, with each factor having three levels, resulting in a total of nine experimental groups. The influence of each factor on tensile strength and extrudability is analyzed using the range analysis method. Ultimately, an optimal formula that meets the multiple requirements of industrial application for tensile strength and other factors is achieved.

1 Experimental part

1.1 Apparatus and reagents used in the experiment

1.1.1 Experimental reagents

The main reagents and suppliers used in this experiment are: 20,000 mPa·s 107 silicone rubberfrom Hubei Shixing Chemical Co., Ltd.; nano-calcium carbonate (with an average particle size of 50~100 nm) from Guangxi Warner New Material Co., Ltd.; methyltrimethoxysilane from Hubei New Blue Sky New Material Co., Ltd.; vinyltrimethoxysilane from Hubei New Blue Sky New Material Co., Ltd.; KH550 from Hangzhou Guibao New Material Co., Ltd.; dibutyltin dilaurate from Hangzhou Guibao New Material Co., Ltd.; silicone oil (350 mPa·s) from Hubei Longsheng Sihai New Material Co., Ltd.

1.1.2 Experimental equipment

The experimental equipment models and suppliers used in this study are as follows: Experimental Dynamic Mixer (QF-5L), Foshan Jinyinhe Intelligent Equipment Co., Ltd.; Electronic Universal Testing Machine (LK-103B), Dongguan Likong Instrument Technology Co., Ltd.; Electronic Analytical Balance (PY-E62), Shenzhen Puyun Electronics Co., Ltd.; Universal Tensile Testing Machine (WDW-1), Shanghai Songdun Machinery Equipment Co., Ltd.; Extrusion Testing Machine, self-made.

2 Experimental design

This experiment investigates the influence of three factors on the tensile strength of silicone rubber through orthogonal experimental design. These factors are the dosage of 107 silicone rubber, the dosage of nano-calcium powder, and the dosage of 350 mPa·s silicone oil. Three levels are designed for each factor (Table 1), utilizing an $L_9(3^3)$ orthogonal experimental design.

Table 1 Design of orthogonal experimental factor levels

Level	107 silicone rubber (A)/%	Mixed nano-calcium (B)/%	Silicone oil (C)/%
1	30	55	3
2	35	60	5
3	40	65	7

Based on the orthogonal table, nine sets of orthogonal experiments were designed, as shown in table 2.

Table 2 Orthogonal experimental design table

Experiment Number	107 silicone rubber(A)	Mixed nano-calcium (B)	Silicone oil (C)
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	3	2

1.3 Sample preparation

Add a certain amount of 107 glue, nano-calcium carbonate, and silicone oil into an experimental dynamic mixer and stir at room temperature for 10 minutes. Then, raise the stirring paddle to scrape off the powder. Ensure that both the stirring paddle and the calcium powder on the cylinder wall are completely scraped off. After scraping, continue stirring, apply vacuum, and turn on the heating device. Once the temperature reaches 110℃, start timing and stir for 3.5 hours. After the stirring time is up, let it stand and cool down. When the temperature returns to room temperature, the base material is obtained.

Add a certain amount of the aforementioned base material and color paste to the experimental power mixer, stir for 5 minutes, and then add the crosslinking agent. Turn on the vacuum pump to evacuate the air and continue stirring for 20 minutes. Finally, add the catalyst, evacuate and stir for another 20 minutes, and then discharge the mixture. Place the resulting paste into a 300 mL glue bottle for later use.

1.4 Test method

1.4.1 Tensile strength and elongation at break

Sample preparation and curing were conducted according to GB/T528-2009. The samples were individually subjected to tensile testing using a dual-servo universal material testing machine, and the force required during the tensile process and the elongation at break were recorded.

1.4.2 Extrudability

The test temperature for the sample is (23±3℃), the diameter of the base hole is 4 mm, and the pressure is set at 0.3 MPa. The volume of extruded silicone rubber is measured for 10 seconds, and the test is conducted three times to obtain an average value.

1.4.3 Surface drying time

Referring to the GB/T13477.5-2017 standard, under a glue application environment of (25±2)℃ and (55±5)%RH, squeeze the glue onto a clean tin foil paper and start timing. Gently touch the surface of the glue strip with the end of your finger, and record the time it takes for the glue surface to become non-sticky.

1.4.4 Curing depth

The maximum depth of vulcanization after testing the sample for 24 hours using a vulcanization depth plate under conditions of (23±2)℃ and (50±5)% relative humidity is measured.

2 Experimental results and analysis

2.1 Analysis of test results

The test results of the orthogonal experiment are presented in table 3.

Table 3 Orthogonal experiment test results

Experiment number	Tensile strength / MPa	Extrusion rate / ($mL \cdot min^{-1}$)
1	2.65	25.4
2	2.50	35.2
3	2.30	52.6
4	2.62	61.2
5	2.51	66.4
6	2.62	56.8
7	2.60	78.6
8	2.63	68.2
9	2.59	61.4

Table 4 calculates the influence weight of each factor on the tensile strength and consistency of the adhesive sample through the range value. The calculation formula is:

$$\omega_i = \frac{R_i}{\Sigma_{j=1}^{n} R_j} \quad (1)$$

R_j—represents the range value of the ith indicator.

Table 4 Analysis of orthogonal experimental data

	107 silicone rubber (A)	Mixed nano-calcium (B)	Silicone oil (C)
	Tensile strength		
k1	2.48	2.62	2.63
k2	2.58	2.55	2.57
k3	2.61	2.50	2.47
r	0.12	0.12	0.16
	Extrudability		
k1	37.73	55.07	50.14
k2	61.46	56.59	52.60
k3	69.40	56.94	65.85
r	31.67	1.87	15.72
	ωi		
Tensile strength	30.33%	29.51%	40.16%
Extrudability	64.29%	3.80%	31.91%

Under the requirements of superior tensile strength and good extrudability, the optimal formula for silicone adhesive is A3B2C1. In terms of tensile strength, the range (R) of silicone oil is the largest compared to the other two factors, indicating that the addition amount of silicone oil has the greatest impact on tensile strength, with an impact weight of 40.16%. According to the range values in the table, the order of influence of the three factors on tensile strength is: silicone oil > 107 silicone rubber > mixed nano-calcium. Similarly, based on the analysis of the range values of extrudability, the order of influence of the three factors is 107 silicone rubber > silicone oil > mixed nano-calcium, with 107 silicone rubber having the greatest impact weight, reaching 64.29%.

Based on the experimental data and analysis above, it can be concluded that the content of silicone oil has a significant and negative impact on the tensile strength of silicone rubber.

The reason lies in the fact that silicone oil, as a small molecule substance that does not participate in the reaction in the system, fills the voids in the polymer network chains. The presence of silicone oil dilutes the effective molecular polymer to some extent, reducing the number of effective polymer chains per unit volume, leading to a decrease in crosslinking density, further loosening of the molecular network, and weakening of the tensile strength. However, the silicone oil molecules themselves are inert, they do not participate in the formation of hydrogen bonds, nor do they competitively bind to the silanol groups on the surface of silica. Their presence, to some extent, increases the average distance between polymer chains, slightly weakens the intermolecular forces (mainly van der Waals forces), and has a negligible dilution effect on the density of the hydrogen bond network. This dilution and lubrication effect does indeed reduce the overall viscosity of the system, manifested as a slight decrease in consistency. However, since it does not affect the fundamental structure of the hydrogen bond network that determines consistency, this reduction effect is limited and mild.

The content of 107 silicone rubber compound has a significant and positive impact on the extrudability of silicone rubber. When the content of 107 silicone rubber compound increases from 30% to 35%, there is a noticeable jump in extrudability. The reason may be that when additional 107 silicone rubber compound is introduced, these newly added long-chain molecules with active silanol groups at both ends competitively adsorb onto the active sites on the surface of silica filler particles. This behavior is equivalent to diluting the originally tightly bridged network nodes with fewer polymer chains and higher strength into nodes with more polymer chains sharing connections and lower strength. As a result, the overall connection strength and density of the entire filler-polymer network are significantly weakened. From a rheological perspective, when this diluted network structure is subjected to shear forces (such as extrusion through a die), it collapses more easily and thoroughly, leading to a sharp decrease in the apparent viscosity of the rubber compound, making extrusion effortless and smooth.

2.2 Market comparison

During the actual construction process, not only must the tensile strength and extrudability of silicone rubber be considered, but its surface drying time and 24-hour curing depth also affect the construction progress during application. Therefore, to verify the feasibility of the formula presented in this article, we selected two commercially available silicone sealants, X1 and X2, for testing. The test results are shown in table 5.

Table 5 Performance comparison between optimized formula and commercial silicone sealant

	Tensile strength (MPa)	Extrusion rate / ($mL \cdot min^{-1}$)	Surface drying time/min	24-hour curing depth (mm)
X1	2.00	31.46	15	3.2
X2	2.52	56.90	20	3.0
Premium Formula	2.63	68.20	10	3.5

The test results in table 5 indicate that, compared with commercially available formulations, the optimized formulation presented in this paper exhibits superior performance and can better meet customer requirements.

3 Experimental conclusions

Through orthogonal experiments, the optimal formula for preparing silicone adhesive was determined as follows: 40% 107 silicone rubber, 60% mixed nano-calcium, and 3% silicone oil. At this point, the tensile strength reached 2.63 MPa, and the extrusion rate was 68.2 mL/min. Compared to commercially available silicone adhesives, the tensile strength, extrusion rate, surface drying time, and 24-hour curing depth of this formula were superior.