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STUDY OF THE CHANGES IN RHEOLOGICAL AND ADHESIVE PROPERTIES THROUGH THE MODIFICATION OF LIQUID GLASS



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Abstract. This paper discusses the development of a modified, environmentally safe adhesive based on liquid glass (sodium silicate $\text{Na}_2\text{O}\cdot n\text{SiO}_2$) with enhanced adhesion efficiency to wood. The sodium silicate used fully complies with the requirements of GOST 13078–81 and TU 6-18-003-87, with a silicate modulus of 2.6–2.8 and a solid residue content of 31–33%. An oligomer based on thiourea and glycerol was used as a modifier. The experiments were carried out by mixing the modifier with liquid glass in various mass ratios (5%, 10%, 15%). The experimental results showed that the modification process significantly improved the rheological and mechanical properties of the adhesive. At the optimal 10% modifier content, the adhesion strength increased from 2.28 MPa to 3.15 MPa (an increase of 27.6%), while water resistance rose from 62% to 82%. The viscosity was approximately 230 mPa·s, which fully meets the industrial range specified in GOST 13078–81 (200–250 mPa·s). The resulting modified adhesive does not release formaldehyde, is non-toxic, and environmentally safe, providing high adhesion and water resistance for wood bonding. The obtained results form a scientific and practical basis for expanding the use of modified sodium silicate-based adhesives in woodworking, construction, and furniture industries.

Keywords: wood adhesive, sodium silicate, bonding strength, curing technology, adhesion, eco-friendly glue, modifier, water resistance.

ИССЛЕДОВАНИЕ ИЗМЕНЕНИЯ РЕОЛОГИЧЕСКИХ И АДГЕЗИОННЫХ СВОЙСТВ ПРИ МОДИФИКАЦИИ ЖИДКОГО СТЕКЛА

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Аннотация. В данной статье рассматривается получение модифицированного, экологически безопасного клея на основе жидкого стекла (натриевого силиката $\text{Na}_2\text{O}\cdot n\text{SiO}_2$) с повышенной эффективностью адгезии к древесине. Используемый натриевый силикат полностью соответствует требованиям ГОСТ 13078–81 и ТУ 6-18-003-87, имеет силикатный модуль 2,6–2,8 и массовую долю сухого остатка 31–33%. В качестве модификатора применялся олигомер на

основе тиомочевины и глицерина. Эксперименты проводились при смешивании модификатора с жидким стеклом в различных массовых соотношениях (5%, 10%, 15%). Результаты экспериментов показали, что процесс модификации существенно улучшает реологические и механические свойства клея. При оптимальном содержании модификатора (10%) адгезионная прочность увеличилась с 2,28 до 3,15 МПа (на 27,6%), а водостойкость — с 62% до 82%. При этом вязкость составляла около 230 мПа·с, что полностью соответствует промышленному диапазону, установленному ГОСТ 13078–81 (200–250 мПа·с). Полученный модифицированный клей не выделяет формальдегид, является нетоксичным и экологически безопасным связующим материалом, обеспечивающим высокую адгезию и устойчивость к влаге при склеивании древесины. Полученные результаты создают научно-техническую основу для расширения применения модифицированных силикатных клеев в деревообрабатывающей, строительной и мебельной промышленности.

Ключевые слова: деревянный клей, натрий силикат, прочность сцепления, технология отверждения, адгезия, экологический клей, модификатор, водостойкость.

SUYUQ SHISHANI MODIFIKATSİYALASH ORQALI REOLOGIK, ADGEZION XOS SALARI O'ZGARISHINING TADQIQI

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Annotation. Ushbu maqolada yog'ochga nisbatan yopishish samaradorligi oshirilgan, suyuq shisha (natriy silikat $Na_2O \cdot nSiO_2$) asosida tayyorlangan modifikatsiyalangan, ekologik xavfsiz elim olish masalasi ko'rib chiqilgan. Ishlatilgan natriy silikat GOST 13078–81 va TU 6-18-003-87 talablariga to'liq javob beradi, uning silikat moduli 2,6–2,8, quruq qoldiqning massaviy ulushi esa 31–33% ni tashkil qiladi. Modifikator sifatida tiomochevina va glitserin asosida sintez qilingan oligomer qo'llanilgan. Tajribalar modifikatorni suyuq shisha bilan turli massaviy nisbatlarda (5%, 10%, 15%) aralashtirish orqali o'tkazildi. Tajriba natijalari shuni ko'rsatdiki, modifikatsiya jarayoni eliminating reologik va mexanik xususiyatlarini sezilarli darajada yaxshilaydi. Modifikatorning optimal miqdori (10%) da yopishish mustahkamligi 2,28 MPa dan 3,15 MPa gacha (27,6%) oshgan, suvga chidamlilik esa 62% dan 82% gacha ko'tarilgan. Shu bilan birga, eliminating qovushoqligi 230 mPa·s ni tashkil etgan bo'lib, bu GOST 13078–81 da belgilangan 200–250 mPa·s sanoat me'yorlariga to'liq mos keladi. Olingan modifikatsiyalangan elim formaldehid ajratmaydi, toksik bo'limgan va ekologik xavfsiz bog'lovchi material hisoblanadi, u yog'ochni yopishtirishda yuqori adgeziya va namlikka bardoshlilikni ta'minlaydi. Olingan natijalar modifikatsiyalangan silikat yelimalarni yog'ochga ishlov berish, qurilish va mebel sanoatida keng qo'llash uchun ilmiy-texnik asos yaratadi.

Kalit so'zlar: yog'och elim, natriy silikat, yopishish mustahkamligi, qotish texnologiyasi, adgeziya, ekologik yelim, modifikator, suvga chidamlilik.

Introduction. At present, the main components of widely used wood adhesives are organic compounds based on urea-formaldehyde, phenol-formaldehyde, and melamine-formaldehyde resins. Such formaldehyde-based organic adhesives hold a dominant position in the field of wood adhesives due to their high bonding properties [1-3]. However, the production, storage, and application

of such formaldehyde-containing organic adhesives continuously release toxic aldehyde compounds, which cause serious harm to all living organisms, including human health [4-5].

Currently, the development of environmentally friendly, non-toxic adhesives that do not harm the atmosphere has become an important research direction in the field of wood

adhesives [6-7].

Inorganic adhesives based on silicates are distinguished by a number of advantages — non-toxicity, high heat resistance, strong bonding strength, low shrinkage stress, and good workability [8–10]. Therefore, they are widely used for bonding materials such as metal, ceramics, glass, stone, and soil [11–12]. Adhesives based on liquid glass (sodium silicate) are applied in various fields, including aerospace, mechanical engineering, electronics, the chemical industry, construction, and many others.

Despite the information mentioned above, the use of silicate adhesives directly for wood is rare because these adhesives are brittle and lack water resistance. Therefore, to adapt silicate adhesives for wood materials, reinforcement through modification is necessary.

In our previous studies, we published our research results on the synthesis of a modifier based on thiourea and glycerol [13]. According to the data presented in scientific articles and dissertations [14–15], properly modified silicate adhesive can be successfully used for bonding wood and is capable of replacing traditional organic adhesives. The incorporation of silicate into wood adhesive, on the one hand, expands the application range of silicate adhesives, and on the other hand, makes it possible to create an environmentally safe adhesive that completely eliminates the problem of toxic compounds released from organic adhesives.

This plays an important role in improving the level of wood adhesive technology and modernizing the wood-based panel industry. The bonding efficiency of adhesives is directly related to the curing technology [16–18]. Therefore, to achieve the highest bonding performance, it is necessary to compare different curing processes and determine the optimal curing technology.

Methods. For the experimental work, we used sodium silicate (liquid glass, $\text{Na}_2\text{O}\cdot\text{nSiO}_2$) as the main binding component. The liquid glass employed fully complies with the requirements of GOST 13078–81 “Sodium Liquid Glass. Technical Specifications.” According to the standard, the silicate modulus ($\text{SiO}_2/\text{Na}_2\text{O}$) of the sodium silicate solution should be within the range of 2.4–3.0, the mass fraction of dry residue should be 25–35%, and the density should be 1.35–1.45 g/cm³. This

normative range ensures the optimal viscosity, ease of processing, and adhesion characteristics of the adhesive composition.

The technical characteristics of the sodium silicate used in our experiments are as follows: silicate modulus 2.6–2.8, mass fraction of dry residue 31–33%, viscosity (at 25°C) 140–160 mPa·s, and pH value 11.0–11.5. Such a composition falls within the requirements of TU 6-18-003-87 “Technical Specifications for Silicate Adhesives and Composite Binders” and provides a stable chemical environment during polycondensation modification processes.

As a modifier, we used an organic composite product synthesized in our laboratory [13]. The synthesis process was carried out in such a way as to maintain chemical stability compatible with the highly alkaline environment of the sodium silicate solution. The modifier contains amino, hydroxyl, and thiourethane groups, which are capable of forming covalent bonds with the $-\text{O}-\text{Si}-\text{O}-$ network of the silicate matrix, thereby providing additional structural strength within the adhesive layer.

During the preparation of the modified adhesive, liquid glass was mixed with the modifier at different mass ratios (5%, 10%, and 15%). The mixtures were homogenized in a mechanical stirrer at a speed of 400 rpm for 15 minutes. The viscosity of the adhesive was determined using a capillary viscometer in accordance with GOST 8420–74 at 25°C, and the curing time was evaluated at 105°C according to GOST 14760–69. The obtained data made it possible to determine the relationship between the amount of active components in the adhesive composition and its viscosity.

To determine the bonding strength, poplar wood specimens with dimensions of 100×20×5 mm were prepared. The wood surface was sanded, cleaned from dust, and the adhesive was applied evenly on both surfaces at a consumption rate of 200–240 g/m². The specimens were tested under three different curing regimes: a) natural curing (at 25°C for 24 hours), b) thermal curing (at 80°C for 2 hours), c) curing under pressure (at 0.75 MPa and 60°C for 1 hour).

The tested specimens were examined using a universal testing machine Instron 3365 in accordance with GOST 14760–69 “Adhesives.

Method for determining the shear strength of bonded joints.” For each regime, five repeated measurements were carried out, and the average value was calculated based on statistical analysis. The bonding strength was determined using the formula $\sigma = F/S$ (MPa), where F is the breaking force, and S is the bonded surface area. Suvga chidamlilik sinovi GOST 12120–76 asosida olib borildi. The bonded specimens were immersed in water at a temperature of 20°C for 24 hours, after which changes in their mass and bonding strength were determined, and the post-immersion strength was evaluated using the formula $R = (\sigma_2/\sigma_1) \times 100\%$.

The rheological behavior of the adhesive was studied using a Brookfield DV-II+ viscometer. Measurements were carried out in the temperature range of 20–60°C at rotation speeds of 5–100 rpm. The obtained flow curves ($\tau-\gamma$) were used to analyze the effect of modification on viscosity and deformation behavior. Statistical analysis was performed using the Statistica 10.0 software, and the reliability of the data was determined based on the Student's t-test at a significance level of $p < 0.05$.

These experimental results make it possible to thoroughly study the physicochemical and rheological properties of liquid-glass-based modified adhesives that comply with the requirements of GOST 13078–81 and TU 6-18-003-87. The obtained types of adhesives are distinguished by being environmentally safe, non-toxic, water-resistant, and having high adhesion to wooden surfaces.

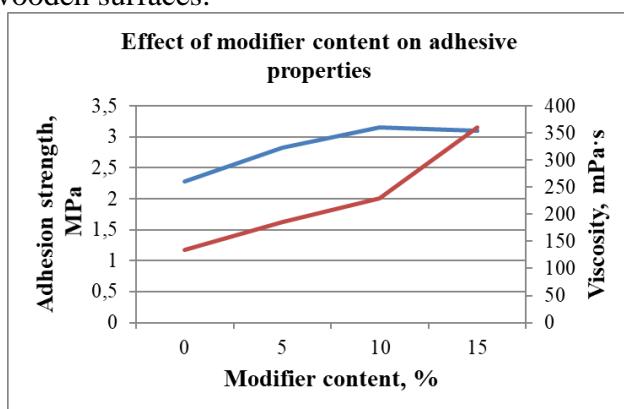


Fig.1. Effect of modifier content on adhesive properties.

Results. The liquid glass ($\text{Na}_2\text{O} \cdot n\text{SiO}_2$) used in the experiments had a silicate modulus of 2.6–2.8. At this modulus value, the $\text{SiO}_2/\text{Na}_2\text{O}$ ratio in the

solution is optimal, maintaining a balance between the stability and reactivity of the silicate network. When the silicate modulus is below 2.4, the solution becomes excessively alkaline, accelerating the hydrolysis process on the wood surface; when it is above 3.0, the solution becomes overly viscous, resulting in decreased adhesion. Therefore, a silicate modulus in the range of 2.6–2.8 was found to be optimal for achieving effective chemical adhesion with wood.

The average viscosity of the unmodified pure silicate adhesive was 135 mPa·s, while its adhesion strength was 2.28 MPa. With the introduction of the modifier, the molecular interactions within the adhesive composition intensified, resulting in positive changes in its rheological parameters. The experimental results are presented in Table 1.

Table 1.
Effect of modifier content on adhesive characteristics

No	Modifier content, %	Curing time, min	Water resistance, %
1	0	35	62
2	5	42	75
3	10	48	82
4	15	65	79

As can be seen from Figure 1 and Table 1, with the increase in the modifier content, both the viscosity and adhesion strength of the adhesive increased to a certain extent. However, when the modifier concentration reached 15%, the viscosity rose excessively (up to 360 mPa·s), resulting in poorer spreadability of the adhesive and uneven curing of the layer. Therefore, a 10% mass fraction of the modifier was determined to be optimal. At this ratio, the adhesion strength of the adhesive reached 3.15 MPa, which is approximately 27.6% higher than that of pure liquid glass.

The water resistance of the adhesive also increased significantly — while the post-immersion strength of the unmodified adhesive was 62%, this value rose to 82% at 10% modification. This indicates that the incorporation of the modifier into the silicate network leads to the formation of new bonds, which reduce the interaction of the adhesive matrix with water molecules.

When the rheological analysis of the adhesive was carried out using a Brookfield viscometer, all samples exhibited non-Newtonian fluid behavior. According to the analysis of the flow curves ($\tau-\gamma$),

the viscosity decreased with increasing shear rate, indicating a pseudoplastic character. This property ensures the easy spreading of the adhesive over the surface and good penetration into the substrate. At 10% modification, the measured dynamic viscosity was 230 mPa·s, which falls within the most favorable range for processing (200–250 mPa·s, in accordance with GOST 13078–81 and TU 6-18-003-87 standards).

Discussion. The above results indicate that the thiourea–glycerol–formaldehyde-based modifier forms chemical bonds within the liquid glass matrix, thereby improving the mechanical and rheological properties of the adhesive layer. The amino and hydroxyl groups of the modifier interact with the –Si–O– bonds of the silicate network through hydrogen exchange reactions, leading to the formation of new –Si–O–C– type bonds. As a result, the plasticity of the adhesive layer increases, microcrack formation is reduced, and water resistance is enhanced.

Most importantly, the resulting modified adhesive does not release formaldehyde, is environmentally safe, and serves as a non-toxic binder that complies with the standards GOST 13078–81 and TU 6-18-003-87. The 10% modification level is recommended as the optimal composition for wood-based materials, as it provides the most balanced ratio of adhesion

strength, viscosity, and water resistance.

Conclusions. As a result of the conducted studies, it was proven that the modification of liquid glass ($\text{Na}_2\text{O}\cdot n\text{SiO}_2$)-based adhesives with organic modifiers significantly improves their physicochemical and rheological properties. The addition of the modifier in a 10% mass fraction increased the adhesion strength of the adhesive from 2.28 MPa to 3.15 MPa, i.e., by 27.6%, while the water resistance increased from 62% to 82%.

Rheological analyses showed that the modified adhesives belong to the class of pseudoplastic non-Newtonian fluids, in which viscosity decreases with increasing shear rate. This property ensures easy spreading of the adhesive over the surface and good penetration into the substrate. At the optimal 10% modification, the viscosity reached 230 mPa·s, which falls within the industrial standard range of 200–250 mPa·s.

In general, the conducted research demonstrates the possibility of creating a new generation of environmentally friendly adhesive compositions through the organic modification of liquid glass. These results have scientific and technical significance for practical applications in the fields of silicate chemistry, materials science, and wood technology.

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