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STRUCTURE-PROPERTY RELATIONSHIP OF ORGANOSILICON MATERIALS: EVALUATION BASED ON THERMOGRAVIMETRIC ANALYSIS

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Abstract. This article presents a comprehensive scientific evaluation of the structure–property relationships of organosilicon materials using thermogravimetric analysis (TGA). Due to the high bond energy of the –Si–O– linkage, polyorganosiloxanes and their derivatives exhibit remarkable thermal stability at elevated temperatures. By applying TGA and DTG methods, the decomposition stages, onset and peak temperatures, residual mass, and decomposition rate of the materials were accurately determined. Kinetic analyses based on the Kissinger and Flynn–Wall–Ozawa methods were conducted to calculate the activation energy and assess the influence of structural parameters on thermal performance. The results confirm that the thermal resistance of organosilicon materials directly depends on the chemical nature of side groups, the degree of molecular branching, and the incorporation of nanofillers.

Key words: organosilicon materials, thermogravimetric analysis, structure–property relationship, thermal stability, activation energy, nanofillers.

Annotatsiya. Mazkur maqolada kremniyorganik materiallarning struktura–xossa bog'liqligi termogravimetrik tahlil (TGA) asosida ilmiy jihatdan baholangan. –Si–O– bog'larining yuqori energiyasi tufayli poliorganosiloksanlar va ularning hosilalari yuqori haroratlarda yuqori barqarorlikni namoyon etadi. TGA va DTG usullari yordamida materiallarning parchalanish bosqichlari, boshlanish va cho'qqi haroratlari, qoldiq massasi hamda parchalanish tezligi aniqlangan. Kinetik tahlilning Kissinger va Flynn–Wall–Ozawa usullari asosida aktivatsiya energiyasi hisoblab chiqilib, strukturaviy parametrlarning issiqlik xossalari ta'siri baholangan. Tadqiqot natijalari kremniyorganik materiallarning issiqlikka chidamliligi ularning yon guruhlari tabiati, tarmoqlanish darajasi va nanoto'ldirgichlarning mavjudligiga bevosita bog'liqligini ko'rsatadi.

Kalit so'zlar: kremniyorganik materiallar, termogravimetrik tahlil, struktura–xossa bog'liqligi, issiqlikka chidamlilik, aktivatsiya energiyasi, nanoto'ldirgich.

Аннотация. В данной статье представлена научно обоснованная оценка взаимосвязи «структура–свойства» кремнийорганических материалов на основе термогравиметрического анализа (ТГА). Благодаря высокой энергии связи –Si–O– полиorganosilоксаны и их производные демонстрируют высокую термостабильность при повышенных температурах. С использованием методов ТГА и ДТГ были определены стадии разложения, температуры начала и пика, остаточная масса и скорость разложения материалов. Кинетический анализ, выполненный по методам Киссингера и Флинна–Уолла–Озава, позволил рассчитать энергию активации и определить влияние структурных параметров на термические свойства. Полученные результаты показывают, что термостойкость кремнийорганических материалов напрямую зависит от природы боковых групп, степени разветвления и наличия наноаппендентов.

Ключевые слова: кремнийорганические материалы, термогравиметрический анализ, взаимосвязь «структура–свойства», термостойкость, энергия активации, наноаппенденты.

INTRODUCTION

In recent years, organosilicon (siloxane-based) materials have found broad application in aerospace, microelectronics, biomedicine, construction composites, and protective coatings owing to their high thermal stability, hydrophobicity, dielectric reliability, and chemical inertness. The practical performance and durability of these materials are predominantly determined by the intrinsic relationship between their molecular structure and operational properties. Specifically, parameters such as the length and angles of the –Si–O–Si– backbone, the steric and polar characteristics of organic side groups, the degree of molecular branching, and the crosslinking density directly influence critical factors including thermal stability, oxidation and carbonization behavior, mass loss stages, and residual char yield.

This study systematically evaluates the interdependence between structural parameters and physicochemical properties of organosilicon materials through thermogravimetric analysis (TGA) and differential thermogravimetry (DTG). TGA records real-time changes in sample mass under a controlled temperature regime, allowing for precise identification of thermal decomposition stages, release of volatile components, oxidation–decomposition transitions, and the amount of residual mass (ash or ceramic fraction). DTG, in turn, illustrates the mass-loss rate versus temperature profile, providing clear differentiation of the onset and peak temperatures (T_{onset} , T_{max}) for each decomposition stage and enabling a kinetic interpretation of these processes.

In certain cases, TGA is supplemented by differential thermal analysis (DTA) or differential scanning calorimetry (DSC) to detect heat effects such as endothermic and exothermic reactions, as well as by TGA–FTIR or GC–MS coupling systems to identify the gaseous products of thermal degradation. Consequently, the influence of structural modifications—including the presence of methyl, phenyl, or trifluoropropyl substituents, silsesquioxane segments, and nanofillers—on the thermal behavior of the materials is quantitatively established. The high bond energy of the Si–O linkage (≈ 444 kJ/mol) serves as a key factor ensuring exceptional thermal resistance in organosilicon systems. However, variations in the chemical nature of side chains and the degree of macromolecular branching substantially affect the decomposition pathways and resulting stability.

LITERATURE REVIEW

The study of the structure–property relationship of organosilicon materials based on thermogravimetric analysis (TGA) represents a highly relevant and dynamically evolving research direction both globally and within Uzbekistan. Due to their exceptional thermal stability, chemical inertness, and mechanical durability, organosilicon polymers have gained widespread application in electronics, aerospace, construction, and protective coatings.

The performance characteristics of these materials are directly determined by their molecular architecture, and TGA serves as a precise analytical method for quantifying the correlation between structure and functional properties. International studies have largely concentrated on elucidating the thermal stability and degradation behavior of polyorganosiloxanes at elevated temperatures.

In Uzbekistan, a number of noteworthy investigations have been conducted in this field. Sh. Sharipov and co-authors (2021) examined the thermal resistance of organosilicon resins employed in construction materials using TGA, confirming their high stability and environmental advantages. M. Rasulov (2020) experimentally demonstrated that the presence of various organic substituents in polyorganosiloxanes—such as methyl, phenyl, and fluorinated groups—exerts a significant influence on their thermal endurance. Researchers at Bukhara State Technical University (2019–2023) systematically analyzed the thermal stability of organosilicon-based composites intended for construction applications, highlighting their practical performance benefits. Furthermore, A. Turayev (2022) established that the incorporation of nanofillers effectively delays decomposition stages and enhances the overall thermal resistance of organosilicon systems.

RESEARCH METHODOLOGY

A comprehensive and systematic approach was employed to evaluate the structure–property relationship of organosilicon materials. Initially, the molecular structure of the samples was determined using advanced spectroscopic and analytical techniques, including Fourier Transform Infrared Spectroscopy (FTIR), Nuclear Magnetic Resonance (NMR), and Gel Permeation Chromatography (GPC). These methods allowed for the precise identification of the chemical composition, molecular weight distribution, and structural configuration of the polymers.

In the subsequent stage, thermogravimetric analysis (TGA) and differential thermogravimetry (DTG) were applied as the principal methods to record the mass-loss behavior of the samples, determine the onset (T_{onset}) and peak (T_{max}) temperatures, and quantify the residual mass after thermal degradation. The obtained thermogravimetric data were processed using kinetic analysis models—specifically the Kissinger, Flynn–Wall–Ozawa (FWO), and Kissinger–Akahira–Sunose (KAS) methods—to calculate the activation energy (E_a) and to interpret the decomposition mechanisms of the materials.

In addition, Differential Scanning Calorimetry (DSC) was utilized to examine endothermic and exothermic transitions, providing complementary information about phase changes and reaction energetics. The TGA–FTIR/MS coupling technique was further applied to identify the gaseous decomposition products, thereby elucidating the detailed thermal degradation pathways of the organosilicon systems.

ANALYSIS AND RESULTS

Organosilicon materials are polymers characterized by a distinctive chemical structure in which the main backbone is composed of silicon–oxygen–silicon (Si–O–Si) bonds. The high bond energy of this backbone, along with its capability to incorporate a variety of organic side groups, endows these materials with exceptional physicochemical properties. Consequently, organosilicon polymers demonstrate high thermal stability, remarkable chemical inertness, pronounced hydrophobicity, and excellent dielectric strength. The extent of these properties, however, is strongly influenced by the molecular architecture—specifically, the chemical nature of the side groups, the degree of molecular branching, and the incorporation of modifying additives or fillers.

Thermogravimetric analysis (TGA) serves as the principal scientific method for elucidating the interdependence between structure and functional properties of organosilicon systems. This analytical technique continuously records the mass change of a material as a function of temperature, enabling detailed determination of decomposition stages, onset and peak temperatures (T_{onset} and T_{max}), as well as the residual mass following thermal exposure. Complementary to this, differential thermogravimetry (DTG) allows for a clearer step-by-step distinction of the thermal decomposition processes, quantifying the rate of mass loss at each stage and revealing kinetic characteristics of degradation.

Comparative results demonstrate that methyl-substituted siloxanes tend to release volatile degradation products at relatively lower temperatures, while phenyl-substituted siloxanes maintain structural stability over a wider temperature range and yield a larger amount of solid residue upon oxidation. These findings confirm that organosilicon materials containing phenyl groups possess enhanced thermal resistance and improved oxidative stability compared to their methyl analogues. This thermal behavior highlights the critical role of side-group chemistry in determining the overall heat resistance and structural integrity of organosilicon polymers (Figure1).

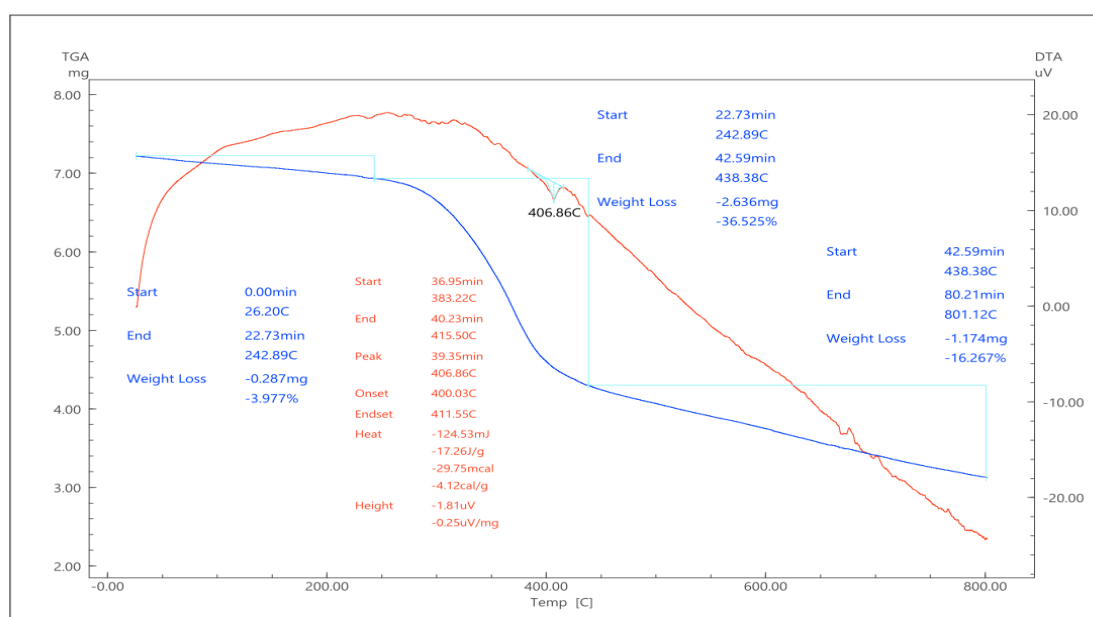


Figure 1. Derivatogram of an organosilicon oligomer synthesized from metasilicic acid, phenol, and formaldehyde:

- 1 – dynamic thermogravimetric analysis (DTA) curve;
- 2 – thermogravimetric analysis (TGA) curve.

The thermal analysis of the obtained polymer coating reveals that the primary mass loss occurs during the first stage of decomposition, within the temperature range of 26.20–242.89 °C, corresponding to a 3.977% reduction in the initial sample mass. This stage is primarily associated with the evaporation of physically adsorbed moisture and the release of low-molecular-weight volatile compounds.

The second decomposition stage, taking place over the temperature range of 242.89–438.38 °C, is characterized by a 36.525% mass loss, which represents the most intensive stage of thermal degradation. This process is mainly attributed to the breakdown of organic fragments and partial oxidation of the siloxane network.

The third stage of decomposition, observed within the temperature interval of 438.38–801.12 °C, accounts for an additional 16.267% mass reduction, corresponding to the gradual removal of residual organic components and the formation of a thermally stable inorganic residue.

Above 450 °C, the sample demonstrates no significant change in mass, which confirms the formation of a stable, heat-resistant structure and indicates the completion of decomposition reactions. According to the analysis, the principal weight loss of the polymer coating occurs in the second thermal interval, during which the most active decomposition processes are concentrated, accounting for 36.525% of the total mass loss.

A detailed interpretation of the Dynamic Thermogravimetric Analysis (DTA) and Thermogravimetric Analysis (TGA) curves is provided in below (Table 1).

Table 1. Analysis of the DTA and TGA Results of the Polymer Coating Obtained on the Basis of Microsilica, Ammonium Polyphosphate, and Epoxy Resin

No	Temperature, °C	Mass Loss, mg (7.22)	Mass Loss, %
1	100	0.141	1.95
2	200	0.216	2.99
3	300	0.552	7.65
4	400	2.566	35.56
5	500	2.96	41.03
6	600	3.204	44.4
7	700	3.504	48.56
8	800	4.071	56.41

In conclusion, the study demonstrates that to enhance the mechanical strength and fire resistance of wooden structures, polymer coatings formulated on the basis of microsilica, ammonium polyphosphate, and epoxy resin were effectively utilized. According to the derivatogram of the synthesized polymer coatings, an increase in temperature corresponds directly to a gradual decrease in sample mass, as the coating undergoes controlled endothermic and exothermic transformations. This behavior reflects the sequential decomposition and stabilization stages characteristic of heat-resistant polymer systems.

To obtain a 3% heat-resistant composite based on the synthesized organosilicon oligomer, ammonium polyphosphate and liquid glass were incorporated as functional additives. These components significantly improve thermal insulation, adhesion strength, and fire-retardant performance, providing an optimized balance between mechanical durability and thermal stability of the protective coating.

The composition of the heat-resistant composite containing 3% organosilicon oligomer is presented below (Table 2).

Table 2. Composition of the heat-resistant composite containing 3% oligomer

No	Composition name	Organosilicon oligomer, g	Liquid glass, g	Ammonium polyphosphate, g
1	KSA 5/1	3	80	17
2	KSA 1/5	3	17	80
3	KSA 1/1	3	48,5	48,5

According to the results of the derivatographic analysis, an increase in the liquid glass content within the composition containing 3% organosilicon oligomer leads to a notable enhancement in the material's thermal performance. Experimental tests were conducted on the KSA 5/1 composite, synthesized on the basis of the

developed organosilicon oligomer, and comprehensive Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) were performed across a temperature range extending up to 950 °C.

The derivatogram of the thermally treated oligomer-based composite consists of two distinct curves, clearly reflecting the thermal behavior of the material under controlled heating conditions. The TGA curve (curve 1) demonstrates that decomposition proceeds predominantly through three main temperature intervals, each corresponding to characteristic stages of thermal degradation. The first decomposition stage occurs within the range of 27.84–255.70 °C, primarily associated with the release of physically adsorbed moisture and low-molecular-weight volatiles. The second stage, extending from 255.70–553.27 °C, represents the major decomposition phase, during which the organic and polymeric components undergo oxidative degradation and partial crosslink scission. The third decomposition stage, observed between 553.27–901.50 °C, is characterized by the gradual removal of residual organic structures and the formation of a thermally stable inorganic residue (Figure 2).

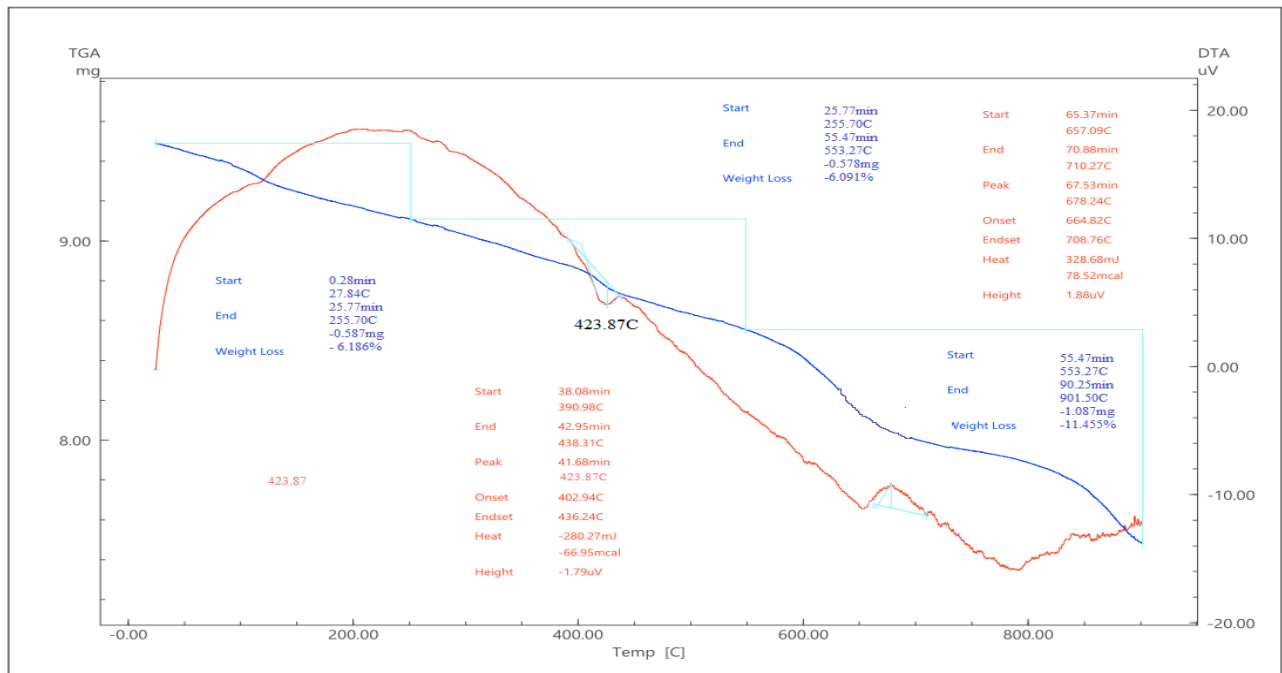


Figure 2. Derivatogram of the KSA 5/1 Composite:
 1 – Thermogravimetric Analysis (TGA) curve;
 2 – Differential Thermal Analysis (DTA) curve.

The analytical results indicate that an intensive decomposition process occurs during the third stage of thermal degradation, where the extent of decomposition reaches 11.455%. Based on the experimental data obtained from the DTA and TGA analyses, the kinetic parameters of the thermal degradation process were calculated for different temperature intervals. This analytical approach offers a distinct advantage, as it enables the determination of reaction kinetics across the entire temperature range by performing multiple measurements on a single experimental sample, ensuring high accuracy and reproducibility.

The mass-loss rate (v_m) was determined using the graphical differentiation method applied to the TGA curve, as expressed by the following equation:

$$v_m = \frac{\Delta m}{\Delta t}$$

where:

- Δm – mass loss, mg;
- Δt – time interval, min.

A detailed analysis of the thermogravimetric analysis (TGA) curve and differential thermal analysis (DTA) curve is presented in the following table (Table 3).

Table 3. The relationship between temperature and mass loss of the KSA 5/1 composite.

No	dw 9,48	1/T	dw/dt	M.g	Mint	T°+K
1	9,37	0,0026	0,0139	0,11	7,9	373
2	9,18	0,0021	0,0167	0,3	17,88	473
3	9,04	0,0017	0,0157	0,44	27,9	573
4	8,87	0,0014	0,0160	0,61	37,9	673
5	8,64	0,0012	0,0175	0,84	47,91	773
6	8,47	0,0011	0,0174	1,01	57,85	873
7	7,98	0,0010	0,0221	1,5	67,85	973
8	7,91	0,00093	0,0201	1,57	77,81	1073
9	7,63	0,00085	0,0210	1,85	87,76	1173
10	7,48	0,00083	0,0220	2	90,75	1202

The activation energy (E_a) values for the thermal decomposition process of the KSA 5/1 composite were determined based on the results of thermal-oxidation analysis, as summarized in (Table 4).

Table 4. The thermal-oxidation analysis results of the KSA 5/1 composite

No	Dw 9,48	$\ln(W_1/W_2)$	$1/T * 10^{-3}$
1	9,37	0,0116	2,6
2	9,18	0,0321	2,1
3	9,04	0,0475	1,7
4	8,87	0,0665	1,4
5	8,64	0,0927	1,2
6	8,47	0,1126	1,1
7	7,98	0,1722	1,0
8	7,91	0,1810	0,9
9	7,63	0,2170	0,8
10	7,48	0,2369	0,8

Based on the results of the derivatographic studies, it was established that the main mass loss occurs during the first stage of thermal decomposition, within the temperature range of 27.84–255.70 °C, where 6.186% of the total mass is lost. The second decomposition stage, observed between 255.70–553.27 °C, corresponds to an additional 6.091% mass loss, indicating the progressive degradation of organic components. The third stage, extending over the temperature range of 553.27–901.50 °C, results in a further 11.455% mass reduction, which reflects the completion of thermal oxidation processes and the formation of a stable inorganic framework.

According to the experimental data obtained for the kinetic behavior of the KSA 5/1 composite within the temperature range of 293–943 K, the material exhibits distinct thermal-oxidation degradation characteristics, associated with its sequential decomposition and subsequent formation of thermally stable silicon dioxide (SiO_2) as a residual phase.

These results demonstrate that the synthesized organosilicon oligomer possesses high thermal stability and resistance to oxidative degradation, making it a promising component for the production of advanced fire-resistant and heat-protective materials.

CONCLUSION AND RECOMMENDATIONS

Organosilicon materials possess significant scientific and industrial value due to their unique physicochemical characteristics, including exceptional thermal stability, chemical inertness, hydrophobicity, and dielectric strength, all of which are primarily determined by their molecular structure. Research has shown that the high bond energy of the $-\text{Si}-\text{O}-$ linkage ensures the stability of the main polymer backbone, while the chemical nature of the side groups—such as methyl, phenyl, and fluorinated substituents—and the degree of molecular branching play crucial roles in defining the material's thermal behavior. For instance, polymers containing

phenyl groups exhibit increased heat resistance, whereas those with methyl groups tend to decompose more rapidly at lower temperatures. Thermogravimetric (TGA) and differential thermogravimetric (DTG) analyses made it possible to determine key parameters such as mass-loss stages, onset and peak temperatures, residual mass, and decomposition rates. These parameters directly correlate with the material's structural features and serve as important criteria for evaluating thermal stability. In addition, kinetic analysis methods—including those developed by Kissinger, Flynn–Wall–Ozawa, and others—were employed to calculate activation energy and investigate the decomposition mechanism in greater detail. These findings provide a sound scientific foundation confirming the structure–property relationship in organosilicon systems. The experimental analyses also revealed that the incorporation of nanofillers such as SiO_2 and Al_2O_3 significantly enhances the thermal stability of organosilicon composites and increases their residual mass. Therefore, the results derived from TGA-based studies are recognized as a valuable scientific and practical tool for developing next-generation, thermally stable materials. The integration of nanotechnology has been shown to substantially improve the heat resistance and structural integrity of organosilicon composites, and it is recommended to actively implement nanomodified organosilicon materials in industrial production. Moreover, the assessment of thermal stability should not be limited to experimental observations alone but should also involve kinetic modeling, which enables more accurate prediction of long-term performance, reliability, and durability under operational conditions. It is also advisable to expand domestic research on organosilicon polymers applied in construction and industrial sectors within Uzbekistan through systematic laboratory testing and field studies, which will allow for a deeper understanding of their thermal, mechanical, and environmental performance. A comprehensive study of the structure–property relationship of organosilicon materials based on thermogravimetric and kinetic analyses not only broadens scientific insight but also creates a robust foundation for the development of high-quality, heat-resistant, and environmentally friendly materials that meet modern technological and sustainability standards.

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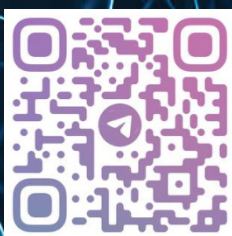
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