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POTENTIAL OF USE THE MODIFIED POLYMERIC SULFUR BASED ON THE BY- PRODUCT PETROLEUM SULFUR IN THE RUBBER PRODUCTION

Abstract. Technological methods have been developed for the production of new polymer sulfur composites based on activated by-product sulfur and polystyrene for their use as a vulcanizing agent with improved binding and astringent properties. Optimum process conditions have been worked out, allowing to vary the sulfur content in the obtained materials. An experimental laboratory batch of new sulfur-polymer composites for testing in extended laboratory conditions as a vulcanizing agent has been gained.

Formulations, optimal conditions for mixing and vulcanization of rubber compounds have been developed. Tests of physical- mechanical and operational properties of rubbers on their basis showed the effectiveness and prospects of their application in the production of tires and rubber products.

The developed technology for the production of new sulfur-based vulcanizing agents based on activated by-product sulfur and polystyrene is promising and can be recommended for use in the production of rubber products with improved performance properties.

Key words: modified polymeric sulfur, polystyrene, rubber, caoutchouc, vulcanization.

Introduction. Vulcanization plays an important role on rubber industry by offering the rubber products containing three-dimensional net work of rubber molecules. By this mean, the significant improvement innumerous properties including tensile and tear properties, set, resilience and abrasion of rubber vulcanizates is resulted. The vulcanization could generally be divided into 3 main systems, i.e., sulfur, peroxide, and metal oxide systems [1-4]. The sulfur vulcanization system is generally preferential because of its superiority in mechanical properties and ease of cure behavior adjustment [5, 6]. Typically, the sulfur used in rubber industry originates from 2 main resources, i.e., natural resource and petroleum refinery. Basically, the sulfur from natural resource is more prefer able because of its certain chemical structure in conjunction with its high sulfur content (99 %). The petroleum-based sulfur is taken as a by-product from petroleum refinery. The annual growth in hydrocarbon production in Kazakhstan and the environmental requirements for the quality of petroleum products resulted in the accumulation of more than 10 million tons of sulfur in the Tengiz field at the sulfur storage site. Our main goal of this work is to make full use of such sulfur by investigating the modification technique for enhancing the petroleum-based sulfur to match the requirement of rubber industry. Thus, the most promising direction for the creation of large-scale production of associated sulfur utilization is the production of polymer compositions of modified sulfur with improved properties for use as vulcanizers and hardeners, as well as a plasticizer of rubber compounds and bases for soft rubbers and elastomers.

This article presents the results of studies on the production of high-sulfur polymers based on the by-produced Tengiz sulfur and polystyrene (PS) and the use of the polymers obtained as the vulcanizing agent of rubber compounds.

Results and discussion. Studies of the using possibility a by-product of oil processing-by-sulfur as a vulcanizing agent of unsaturated rubbers compared to commercially available rhombic sulfur offer several advantages. In contrast to conventional polymeric sulfur has a complex of unique properties: high resistance to aggressive media, high impact strength, the absence of thermal shrinkage deformations in the compositions, insolubility in organic solvents and rubbers, does not "bloom" on the surface of rubbers and vulcanizates, which stabilizes the properties of the product in for long periods of storage and can be widely used as a vulcanizing agent in the tire and rubber industries [7-10].

At this study, the production of polymer sulfur was carried out by direct catalytic sulphurization of polystyrene (PS) using by-product sulfur. In order to obtain high-sulfur polymers, deep sulfurization of granular polystyrene by Tengiz sulfur was carried out, and the physical-chemical properties of the products obtained were studied. In the synthesis, activated colloidal nanosized sulfur was used, obtained by acid decomposition of calcium polysulphides [8].

The reaction was studied in the temperature range 240-330 °C at a molar ratio of PS and sulfur (16 grams of sulfur atoms per unit PC), calculated on partial or exhaustive sulfurization. The synthesized samples are powders of dark gray and black color with metallic sheen, insoluble in water and organic solvents.

The synthesized samples were analyzed for the content of S (C, H). According to the thermogravimetric analysis, the polymers obtained are heat-resistant up to 220-240 °C.

Copolymer sulfur modified with polystyrene (SPSM-PS) is formed by the catalytic copolymerization of colloidal sulfur, obtained on the basis of by-product sulfur (by using diphenylguanidine, DPHG, as a catalyst). It was expected that the reaction would proceed according to figure 1 [11-13].

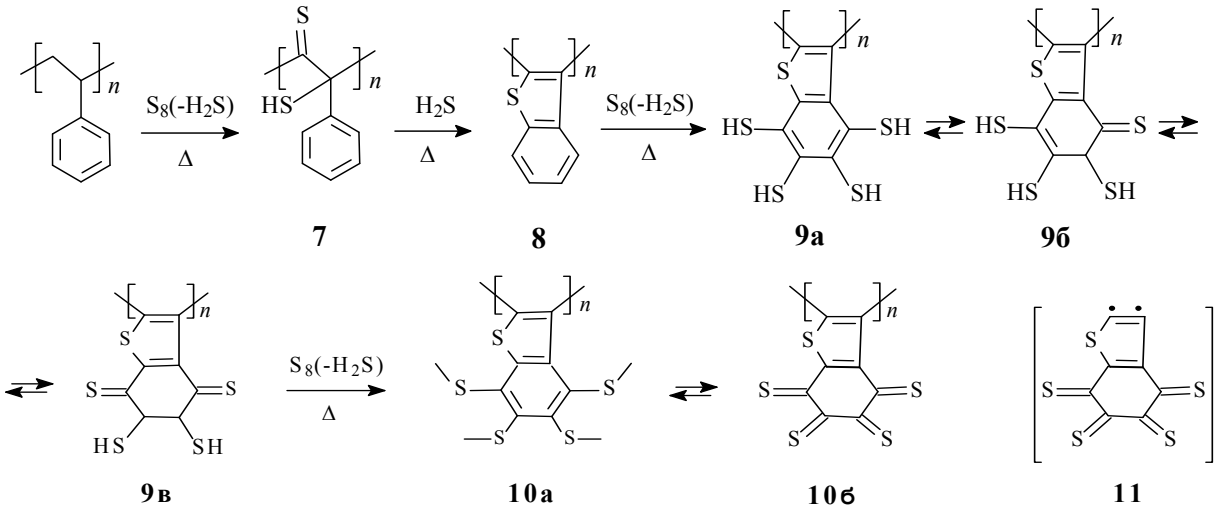

Figure 1 – Reaction scheme of copolymer sulfur modified with polystyrene

As can be seen from the scheme, in the copolymerization products there are condensed benzothiophenes linear and partially cross linked polymers with sulfide units. Under the studied conditions, high-sulfur polymers are formed from PS and colloidal sulfur (content S = 65.7-73.0%) (table 1).

Table 1 - Results of catalytic sedimentation of PS by elemental sulfur

Experi-ence	PS:S, mol	T_a, °C	Time, h	Catalyst, wt. %	Yield	S, %
1	1:8	240	2	DPHG(1)	47.0	42.1
2	1:16	240	2	DPHG (1)	78.1	66.5
3	1:16	240	3	DPHG (1)	98.1	73
4	1:16	300	3	no	96.5	65.7

Analysis of the data given in table 1 shows that the molar ratio of polystyrene to sulfur of 1:16 is optimal, because the molar ratio of PS:S = 1:8 shows a low yield of the product and a low sulfur content in it (runs 1-4). It should be noted that the catalytic synthesis of the sulfurized polystyrene with activated colloidal by-product sulfur shows the best yields (98.1 %) and sulfur content (73 %, experiment 3) compared to elemental sulfur (experiments 1, 2).

In the IR spectra of the compounds obtained (figure 2), the absorption peak with a maximum at 471 cm^{-1} refers to the ν (S-S). Absorption in the 1700-1000 cm^{-1} region (maxima of 1639-1403 cm^{-1}) is apparently due to the vibrations of the skeleton of the exhausted sulfurized benzothiophene skeleton 10a (figure 1). The peak of 1108 cm^{-1} can be attributed to the oscillations of ν(C = S) in structures 9 b, c and 10 b. The triplet 757, 729, 707 cm^{-1} may be due to the mixed vibrations of the aromatic bonds C-C and C-S, in this case in the structure 10 a.

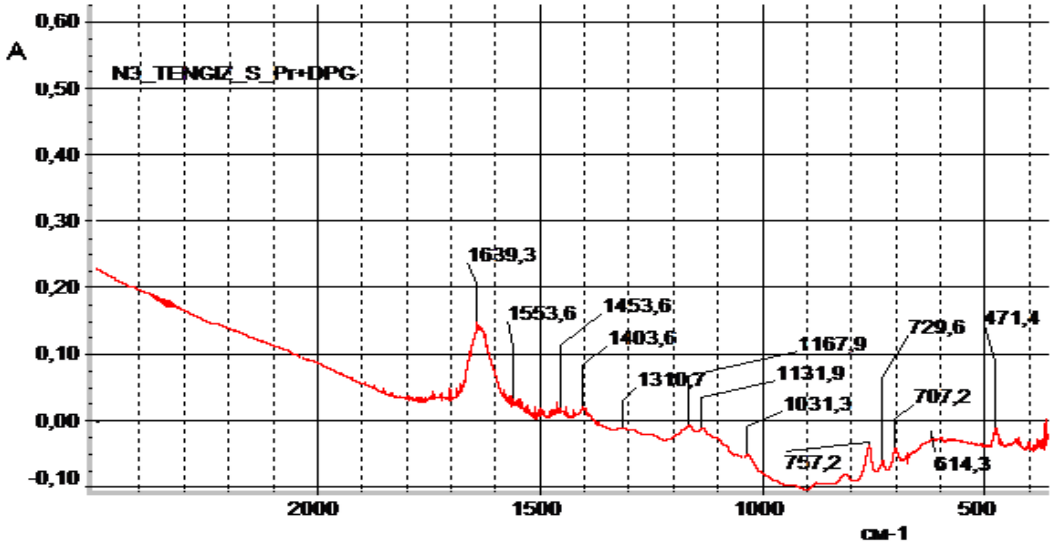

Figure 2 – IR spectrum of sulfurized polystyrene

Development of formulations, optimal conditions for mixing and vulcanization of rubber compounds. The resulting high-sulfur polymers on the basis of associated sulfur and polystyrene were tested during vulcanization of rubbers.

The tests were carried out in the enlarged laboratory conditions on the serial formulations of rubber compounds for the insulation of the bead wire of truck tires containing various amounts of high-sulfur polymer.

The necessary amount of colloidal sulfur preparation depends to a large extent on the compounding formula. As a rule, with an increase in its content for the same composition in the rest of the mixture, the degree of vulcanization increases to a certain optimum. With a further increase in the sulfur dosage, hardness continuously increases, but the overall technological properties deteriorate. For the production of rubber products, 0.2-5.0 wt. h. sulfur (based on 100 parts by weight of rubber); Conversely, for ebonite mixtures, the sulfur content must be increased to 25-40 wt.h. The dosage sulfur in rubber mixtures depends on the rubber type and the number of vulcanization accelerators taken. In mixtures based on sodium-divinyl rubbers, sulfur is taken in an amount of up to 2.5 %, in mixtures with divinyl-styrene rubbers up to 2 %. Rubber is a complex multicomponent system, consisting of 10-15 or more ingredients.

SKMS-30 ARKM-15 (GOST 11138-78 E) - synthetic methylstyrene rubber with a methylstyrene content of 30 %. This rubber refers to the "oil" with an average content of highly aromatic oil. Properties of butadiene-methylstyrene rubbers SKMS largely depend on the content of α-methylstyrene in them.

To establish the curing activity of the obtained SPSM-PS, laboratory samples of copolymer sulfur were tested on model filled rubber compounds based on SKMS-30 ARK rubber of insulation of the bead wire in comparison with the standard (table 2), where a different content of the modified polymer sulfur SPSM- PS (2, 3, 4, 6 mph).

Table 2 - Formulation card. Cipher 3 - 111. Assignment: Insulation of the bead wire

	At 100 m.ch. rubber, m.ch.				
	Standard	1 var	2 var	3 var	4 var
SKMS-30 ARC	70,0	100,0	100,0	100,0	100,0
SKI-3 2 gr.	30,0				
Technicalsulfur	4,0				
Sulfur modified polymeric		2 m.ch.			
Sulfur modified polymeric			3 m.ch.		
Sulfur modified polymeric				4 m.ch.	
Sulfur modified polymeric					6 m.ch.
Sulfenamide M	1,2	1,2	1,2	1,2	1,2
Santograd	0,2	0,2	0,2	0,2	0,2
ZincWhite	4,0	4,0	4,0	4,0	4,0
Stearicacid	2,0	2,0	2,0	2,0	2,0
Pinerosin	6,0	6,0	6,0	6,0	6,0
Softener ASMG	7,0	7,0	7,0	7,0	7,0
Oil PN - 6SH	4,0	4,0	4,0	4,0	4,0
Technical carbon P-514	70,0	70,0	70,0	70,0	70,0
Total	198,4	197,4	195,4	198,4	200,4

As the temperature and degree of vulcanization increase, the solubility of sulfur in rubber increases significantly. In natural rubber, during the mixing process at a temperature of 55-56 °C, its solubility reaches 3-4 % of the mass of the rubber.

In the production of soft rubber, where the sulfur content usually does not exceed 3%, during the mixing of the rubber compound, all sulfur can dissolve in the rubber. At the vulcanization temperature, the solubility of sulfur reaches 10%.

When the rubber mixture is cooled, supersaturated solutions can form, from which, due to diffusion, excess sulfur partially crystallizes onto the surface of the rubber mixture. Such a crystallization of sulfur on the surface of a rubber compound or vulcanizate is called "blooming" of sulfur.

Crystallization of sulfur on the surface of rubber unvulcanized parts reduces stickiness, which causes difficulties in assembling rubber products. Reduction of sulfur fading is observed when:

1) introduction of some softeners (stearic acid and pine resin into the rubber mixture, obviously, because these softeners are sulfur dispersants that promote a more even distribution of it in the rubber compound;

2) introduction of a regenerate and carbon black into the rubber mixture, which is explained by the high solubility of sulfur in the regenerate and the adsorption of sulfur by a gas black;

3) conducting the mixing process at the lowest possible temperatures, at which part of the sulfur remains in the undissolved state;

4) the use of small amounts of sulfur during vulcanization, as well as the use of mixtures of sulfur with selenium. The fading of sulfur in rubber is one of the signs of the rubber's lack of vulcanization, and also the consequence of the excess content of free sulfur in rubber. Mixing of rubber compounds was carried out in two stages on laboratory rollers PD 320 160/160. The temperature of the front rollers is 50-60 °C, the rear temperature is 60-70 °C. Duration 25 minutes in the first stage; on the second - 6 min. Vulcanization of the samples was carried out on a vulcanizing press RDE 800x800, at a temperature of 1550 °C, for 20 minutes (table 3). Difficulties were not observed during the mixing process, high-sulfur polymers are easily introduced into the rubber mixture. The distribution of polymer sulfur in rubber is satisfactory, which does not require a change in the rolling and vulcanization regime. The production of a rubber compound for the insulation of the bead wire of trucks was carried out in the following mode (table 3):

Equipment: Laboratory rollers PD 320 160/160

Roller temperature: Front 50-60 °C; Rear 60-70 °C

Table 3 - Mode of manufacturing of a rubber compound for insulation of the bead wire of truck tires

Name of operation	Time, min	
	Beginning of operation	End of operation
1 mixing stage		
Loading of rubbers, SKMS-30, ARC, SKI-3, plasticization	0	3
Load loose ingredients (zinc white, pine rosin, stearic acid, santograd, softener ACMG) and 1 / part of the carbon tetrachloride P-514, mix, rolling rolls on both sides 8 times (4 times on the left side, 4 times on the right)	3	8
Cut 1/3 part of the mixture, load 1/2 part of technical sulfur P-514, insert the cut 1/3 part of the mixture, mix, rolling rolls on both sides 8 times (4 times on the left side, 4 times on the right)	8	12
Cut 1/3 part of the mixture, load the plasticizer PN-6SH, load 1/2 part of the technical sulfur P-245, insert the cut 1/3 part of the mixture, mix, roll rolls on both sides 8 times (4 times on the left side, 4 times with the right)	12	17
Table 3 continue		
Cut 1/3 part of the mixture, load 1/2 part of technical sulfur P-245, add cut 1/3 of the mixture, mix, roll rolls on both sides 8 times (4 times on the left side, 4 times on the right)	17	23
Remove the mixture from the rollers	23	25
Total		25
2 mixing stage		
Load the mixture of the first stage, heat	0	2
Cut 1/3 part of the mixture, load the Sulfenamide M acce-lerator, insert the cut 1/3 part of the mixture, mix, rolling rolls on both sides 4 times (2 times on the left side, 2 times on the right side)	2	4
Cut 1/3 part of the mixture, load the modified sulfur, insert the cut 1/3 part of the mixture, mix, rolling rolls on both sides 4 times (2 times on the left side, 2 times on the right)	4	5
Remove the mixture from the rollers	5	6
Total		6

After the first operation, the rubber mixture was aged for at least 2 hours, the mixture was cooled to room temperature not higher than 250 °C. Vulcanization of the plates was carried out at a temperature of 1550 °C for 20 minutes. At the same time the equipment was used: vulcanization press RDE 800x800.

Thus, as a result of the research, new high-sulfur polymers based on colloidal Tengiz sulfur and polystyrene have been synthesized.

Technological parameters of obtaining sulfur-enriched polymeric composites have been worked out.

It is shown that polystyrene is sulfurized by excess sulfur at temperatures of 240-330 °C. Conditions for carrying out the reaction have been found, which make it possible to vary the sulfur content of the obtained materials. At the same time, the product contains 63.7-73% sulfur.

The resulting composites have been studied as a vulcanizing agent for rubber compounds. Developed recipes, the optimal conditions for mixing and vulcanizing rubber compounds.

In accordance with the developed prescription map (table 3), high-sulfur polymer compositions based on associated sulfur and polystyrene on the physico-mechanical properties of rubbers as a result of vulcanization were also studied.

To establish the curing activity of copolymer sulfur modified with polystyrene-SPCM-PS, laboratory samples of copolymer sulfur were tested on model filled rubber compounds based on SKMS-30 ARK rubber on board wire insulation in comparison with the reference (table 2), where different technical contents were used instead of technical sulfur modified polymer sulfur SPSM-PS (2, 3, 4, 6 m.ch).

Properties of rubber compounds and vulcanizates based on rubbers of general purpose for manufacturing insulation of bead wire of truck tires with additives of polymer sulfur are given in table 4.

Calculation of rheometric curves recorded on a Monsanto rheometer given in table 5.

Table 4 - Physical and mechanical properties of rubbers using modified sulfur

Name of indicators	Control standards	Standard of sulfur tech-4m.ch.	Modified sulfur				
			1 var 2 m.ch	2 var 3 m.ch.	3 var 4 m.ch.	4 var 5 m.ch.	5 var 6 m.ch.
Plasticity, conv. units	0,23-0,33	0,30	0,37	0,34	0,31	0,30	0,34
Breaking strength, kgf / cm^2	n/m 112	140	120	120	135	140	140
Relative elongation,%	180-350	345	367	367	350	355	335
Tear resistance, n / cm	n/m 60	65	55	55	62	63	63
Hardness, conv. units	65-75	71	65	65	67	68	70

Table 5 - Calculation of rheometric curves recorded on a Monsanto rheometer. Mode: 1550C x 20 minutes

Indicator name	Standart	1 variant	2 variant	3 variant	4 variant	5 variant
M_1 τ_1	10 units 1'22"	damp	10,5 units 1'35"	11units 1'25"	11 units 1'25"	12 units 1'22"
M_2 τ_2	12 units 2'15"	-	11,5 units 2'20"	11,5 units 2'20"	11,5 units 2'20"	12 units 2'15"
M_3 τ_3	35,2 units 18'00"	-	36,1 units 17'30"	36,2 units 17'20"	36,2 units 17'20"	37,2 units 17'10"
M_4 τ_4	38 units 19'15"	-	39 units 19'20"	39 units 19'20"	39 units 19'20"	40 units 19'27"

The positive influence of the investigated high-sulfur polymers on the technological properties of the obtained rubber mixtures was established. The results given in Table 4 indicate that the use of polymer-modified polymeric sulfur as the vulcanizing agent improves such physico-mechanical characteristics of rubber products as plasticity, elongation and hardness.

The tests carried out showed that, compared to the standard sample, the plasticity index at a polymer sulfur content of 3 parts by weight are improved by 23 %, a further increase in the content of polymeric sulfur to 6 m.ch. leads to a decrease in this indicator. The value of the relative elongation is maximum at a content of 3 m.p. polymeric sulfur, an improvement of 22 % of the standard sample. The optimum sulfur content is 3-4 parts by weight by 100 parts by mass. rubber.

Conclusion. Thus, based on the results of the conducted studies, it can be concluded that the use of polymeric sulfur as a curative agent has a positive effect on the properties of rubbers based on SKI-3, SKMS-30, ARKM-15.

Based on the results of enlarged laboratory tests on the use of polymer sulfur obtained during the vulcanization of rubber compounds, optimum amounts of polymer sulfur were determined to produce vulcanizates with the best performance properties.

The developed technology for the production of new sulfur-based vulcanizing agents based on activated by-pass sulfur and polystyrene is promising and can be recommended for use in the production of rubber products with improved performance properties.

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МҰНАЙ-ГАЗ ӨҢДЕУІНІҢ ІЛЕСПЕЛІ КҮКІРТ НЕГІЗІНДЕ АЛЫНҒАН ПОЛИМЕРЛІ КҮКІРТТІҢ РЕЗЕҢКЕ ӨНДІРІСІНДЕ ҚОЛДАНУ МҮМКІНДІКТЕРІ

Аннотация: Белсендірілген ілеспелі күкірт пен полистирол негізінде өңделген, олардың ары қарай жақсартылған байланыстырушы және тұтқырлаушы қасиеттері бар вулканизацияланатын агент ретінде пайдалану үшін полимерлі күкірттің жаңа композицияларының технологиялық әдістерінің өндірісі дайын-далды. Алынған материалдардың күкірт құрамын өзгертуге мүмкіндік беретін оңтайлы технологиялық шарттар жасалды. Вулканизацияланған агент ретінде кеңейтілген зертханалық жағдайларда тестілеу үшін жаңа күкірт-полимерлік композиттердің эксперименталды зертханалық партиясы әзірленді.

Оның негізінде құрамдар, резеңкелі қосылыстарды араластыру және вулканизациялаудың оңтайлы шарттары жасалды. Резеңкелердің физика-механикалық және пайдалану қасиеттерін сынау олардың негізінде шиналар мен резеңке бұйымдарын өндіруде қолдануды одан әрі зерттеудің тиімділігі мен келешегін көрсетті.

Белсендірілген ілеспелі күкірт пен полистиролға негізделген жаңа вулканизацияланатын заттарды өндірудің дамыған технологиясы келешекті болып табылады және жақсартылған пайдалану сипаттамалары бар резеңке өнімдерін өндіруде пайдалануға ұсынылуы мүмкін.

Тірек сөздер: жетілдірілген полимерлі күкірт, полистирол, резеңке, каучук, вулканизация.

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ВОЗМОЖНОСТИ ИСПОЛЬЗОВАНИЯ МОДИФИЦИРОВАННОЙ ПОЛИМЕРНОЙ СЕРЫ НА ОСНОВЕ ПОПУТНОЙ НЕФТЕГАЗОВОЙ СЕРЫ В ПРОИЗВОДСТВЕ КАУЧУКА

Аннотация: Разработаны технологические методы производства новых композиций полимерной серы на основе активированной попутной продукции серы и полистирола для их использования в качестве вулканизирующего агента с улучшенными связующими и вяжущими свойствами. Разработаны оптимальные условия процесса, позволяющие варьировать содержание серы в полученных материалах. Наработана экспериментальная лабораторная партия новых серно-полимерных композитов для испытаний в расширенных лабораторных условиях в качестве вулканизирующего агента.

Разработаны составы на их основе, оптимальные условия для смешивания и вулканизации резиновых смесей. Испытания физико-механических и эксплуатационных свойств каучуков на их основе показали эффективность и перспективы их дальнейших исследований для применения в производстве шин и резиновых изделий.

Разработанная технология производства новых вулканизующих веществ на основе активированной попутной серы и полистирола является многообещающей и может быть рекомендована для использования при производстве резиновых изделий с улучшенными эксплуатационными характеристиками.

Ключевые слова: модифицированная полимерная сера, полистирол, резина, каучук, вулканизация.

МАЗМҰНЫ

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