

The Effect of Silica Filler Source on the Mechanical Properties of Composite Rubber

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Abstract. Rubber composite is a high molecular weight polymer, produced from natural or synthetic rubber with sulfur as its common crosslinking agent. To improve the mechanical properties of rubber, filler with high silica content was usually added. Hence in this experiment, due to their high silica content, Risk husk ash (RHA), along with chemizil and zeosil were used as the composite filler. Sulfur was chosen as the crosslinking agent of a rubber mixture that consists of polyisoprene rubber (IR), acrylonitrile butadiene rubber (KNB), and polybutadiene rubber (BR). The composition of the other component was fixed at Per Hundred Rubber (PHR) of 51.43. During mixing, the temperature and mixing time were kept constant at 60-70°C and 6 minutes. The properties of rubber composite were characterized based on their physical and mechanical properties. Based on the rheological test, RHA has a faster vulcanization time compared to chemisil and zeosil. Improvement of the mechanical properties of the RHA rubber mixture was also observed, with 300% modulus, elongation, and tear at 42 kg/cm², 867%, and 51 kg/cm². Meanwhile, insignificance differences in tensile strength, hardness, and specific gravity of RHA compared to chemisil and zeosil, were observed.

1 Introduction

Fillers in rubber blending have many advantages, such as increasing physico-mechanical properties (tensile strength, hardness, tear strength, and elongation) and reducing material costs during the process [1]. A silica which consists of silicon dioxide (SiO₂) is a non-black filler material with a strengthening capability that is proportional to carbon black. Silica provides improvements in heat accumulation, durability, mechanical properties, and so on. The commercial silica (SiO₂) which used in industry are Chemisil, Si-69, precipitated SiO₂, Zeosil, and others [2]. However, the carbon black or silica production process usually requires large costs, because it consumes high thermal energy, so that process is not environmentally friendly [3].

Several studies have been carried out to replace filler using silica from natural materials, one of them, making natural rubber compound from coal briquette ash filler and palm shell charcoal, the result was that rubber compound with palm oil shell charcoal filler material produced better physical properties than coal briquette ash. With a breaking stress value of around 134 kg/cm², an elongation at a break of 710%, and a specific gravity of 1.14 gr/cm³. Rubber compound with coal briquette ash filler produces a hardness value of 47 shore A, better tear resistance of 21.1 kg/cm², and the optimum maturity time is faster than palm shell charcoal. The weakness of this research was that it still used a mixture of carbon black as a filler with a ratio of 20 PHR carbon black: 20 PHR natural filler. Therefore, further research was needed to

review the composition and formulation of the usage of alternative fillers following the required quality requirements [4].

The research using palm shell charcoal with 40 PHR as a rubber compound filler, the result of that research showed the three best values for hardness, namely 58 Shore A, breaking stress 208 kg/cm², and elongation at break, namely 610% [5].

A mechanical analysis of rubber compounds using palm oil boiler ash nanoparticles (49.2% silica). The results of that research obtained the best composition of the compound with nanofiller particles of palm oil boiler ash, namely 8% by weight. The tensile strength value was 17.33 MPa, elongation at break 150%, and hardness 55 Shore A [6]. Based on (SNI 06-1542-2006) the tire compound in motorized vehicles must have a value of 50±5 (Shore A). According to (SNI 12-0172-1987) the decision extension value for shoe soles was at least 100%. And for shoe outsole compounds, at least 100% (SNI 12-0172-2005).

A research shows the effect of a mixture of silica filler and blood-clam shell on the mechanical properties of natural rubber shoe sole compounds. The best physical and mechanical properties of vulcanized sports shoe soles based on SNI 06-1884-1990 Ed.1.2 were samples with a composition of 30 PHR silica filler and 30 PHR clam shell that had a hardness of 53 Shore A, the tensile strength of 18.6276 N/m², tear strength of 12.7452 N/mm² and abrasion resistance of 439 mm³[7]

The research on the usage of coconut fiber as a reinforcing filler in the manufacture of ebonite rubber, the

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results of that research obtained the best characteristics of coconut fiber filler at 45 PHR with a hardness value of 82 (Shore A), stress at break of 6.8 MPA, elongation at break of 40 % and tear resistance of 60 N/mm² [8].

Rice husks are agro-industrial waste, especially in rice production. The usage of rice husk ash was established several years ago for agriculture, bio-mass fuel, and polymer applications. For example, Rice Husk Ash (RHA) has been used as a filler for polymer applications, namely white RHA and black RHA. Apart from that, in terms of carbon content and the effect of its addition to rubber vulcanization, white RHA also has almost the same properties as commercial carbon black [2].

The previous research was conducted using Silica from rice husk ash and compared it with zeosil, the results obtained from that research, allowed us to conclude that the usage of RHA in tread compounds significantly reduced strengthening compared to commonly used filler materials: carbon black and silica. The result showed that RHA could reduce tensile strength, elongation, and tear resistance [4]. Rahmaniar and Susanto (2019) [1] conducted research using silica from rice husk ash as a filler for NR and EPR, the results showed us that the addition of RHA to NR composites with and without EPR caused a slight decrease in tensile strength, elongation at break, and compression set. On the other hand, the hardness of the composite tended to increase due to the addition of RHA [1].

The previous research which was studied the quality of rubber composites filled with rice husks and compared them with two other commercial reinforcing fillers, which were Hi-Sil silica. 233 and carbon black N330. Their observations showed that increasing the rice husk ash filler load from 0 to 40 PHR (Per Hundred Rubber) could help to provide high hardness but low tensile strength and low tear strength, the viscosity and hardening time of the rice husk ash filler rubber also decreased [9]. Only better elasticity properties were observed in the products, compared to those filled with silica or carbon black. Based on the results above, we can conclude that rice husk ash can be used as a low-cost filler in the rubber industry at a time when improving mechanical properties is not considered that important.

In this research, RHA silica was used as a filler in making rubber because of the abundant availability of rice husk waste in the environment, so it could be one way of utilizing waste. Apart from that, silica from RHA is also considered more environmentally friendly than carbon black. In this study, the number of PHR (Per Hundred Rubber) was fixed at 51.43. Then, the mechanical properties of rubber made from RHA were compared with rubber using commercial silica fillers (zeosil and chemisil). The analysis process of the mechanical properties of rubber compounds using rheology, hardness, tensile strength, elongation at break, tear-resistance, and breaking-resistance.

2 Materials dan Methods

2.1. Materials

The three types of elastomers mixed inside the Banbury machine, namely IR (10 PHR), KNB (10 PHR), and BR (80 PHR). Whereas chemisil, zeosil, and RHA mixed at 51.43 PHR. The other materials composition correspondingly mixed in the Banbury machine based on the US Patent No. 9833037. The accelerators, such as TBzTD and MBTS, with Zinc Oxide as activators (4.8 PHR), and Soybean Oil (1.0 PHR), were also added to the Banbury machine. Polydisulfide was chosen as the curing agent of the elastomer mixture. All the chemicals used in this research were industrial grade.

2.2 Methods

The compounding material is based on the American Society for Testing and Materials (ASTM-D3184-80). The mixing process of elastomers was done in a Banbury machine with the following stages:

1. The chemicals from each compound formula were weighed according to a determined formula. The amount of each ingredient in the compound formula was stated in PHR (weight Per Hundred Rubber) regarding the conversion factor.
2. After that, take the BR plastic wrap, put IR (10 PHR), KNB (10 PHR) and BR (10 PHR) and Soybean oil in the Banbury, and make sure the temperature of the Banbury reaches 60-70°C before adding the polymer, Mixing time started at minute 1 when all polymers were put in and when the ram/pounder was going down.
3. Then, add ½ zeosil/chemisil/RHA ZnO filler, and steric acid at the start of the mixing time of 2 minutes when the polymers were all in and when the ram/mixing tool was going down.
4. Next step, clean it with a broom, then add the rest of the chemicals, such as titanium or zirconium compounds, antioxidants, antiozonants, and UV inhibitors, starting with a mixing time of 3 minutes when the polymer is all put in and when the ram/molder is going down.
5. At the end, dump or measure the dump's temperature of the component materials.
6. Separated it, before softening process of NR (IR) and NBR using a roll/Banbury machine for NR(IR) and NBR's mastification until it looked shiny and had fewer holes, duration/cycle can be determined by the factory based on the type of roll/banbury machine, need to be kept for 24 hours after mastification process before proceeding to the next step.

The vulcanization process is done in an open mill with the following steps:

1. Put the material into the open mill at a constant temperature of 60-70°C, then roll it for 2 minutes.
2. Added activator (40% active PHR), accelerator (75% active PHR), and sulfur (75% active PHR) as hardening agents. The same precise amounts of activator, sulfur, and accelerator, were used in the three rubber mixtures, then the rolling process for 15 minutes.

3. Further vulcanization process is done in a hot press machine, which operated at a temperature of $150^{\circ}\text{C} \pm 5^{\circ}\text{C}$ with a curing time of 330 ± 30 and a pressure of 150 ± 10 psi.

Evaluating the properties of the result composite rubber, several characterizations were carried out, which were Tensile Strength, Elongation, Tear Strength, Hardness, and Abrasion.

2.2.1 Rheology testing

The sample preparation for rheology testing involved shaping the material into specimens using circular molds with a component thickness of ± 5.5 mm. The testing was conducted at a temperature of 150°C for 15 minutes. The percentage of reversion at each curing time (t) was calculated using the formula (Tamási & Kollár, n.d.) in accordance with the ISO 3417 method [9].

$$\%Reversion = \frac{(T_{max} - T_t)}{(T_{max} - T_{min})} \times 100 \quad (1)$$

2.2.2 Mechanical properties

The materials tested were molded using specimens that comply with SNI 2019.

Tensile Strength

The results obtained from the tensile strength test as contained in ASTM D-412-16 and calculated using the following equation:

$$Tensile\ strength = \frac{L}{t \times w} \quad (2)$$

Elongation

The results obtained from the elongation test as contained in ASTM D-412-16 and calculated using the following equation :

$$Elongation\ (\%) = \frac{(D2 - D1)}{D1} \times 100 \quad (3)$$

Modulus

The Modulus 300% (kg/cm^2) determined by the force required to stretch the test specimen from the initial gauge length of 2.5 cm to a length of 10.0 cm at the testing area as contained in ASTM D-412-16. Calculate the Modulus 300% as follows:

$$300\% \text{ Modulus} = \frac{s}{t\ (\text{cm}) \times w\ (\text{cm})} \quad (4)$$

Specific Gravity

Specific Gravity measurement was conducted based on ASTM D-297-15. The results obtained from this test were calculated using the following equation:

$$S.G = \frac{W_a\ (g)}{W_a\ (g) - W_w\ (g)} \quad (5)$$

Hardness

Hardness testing conducted in accordance with the ASTM D-2240-97 standard. Hardness is measured using a Shore A durometer (RX-DD-4, Electromatic, NY, USA) with an

accuracy of 0.1 Shore A. For each specimen, hardness is measured at four different locations, from which the average Shore A hardness is determined.

Abrasion

The objective of this test was to evaluate the abrasion resistance of solid elastomers and foams using the Akron abrasion device. To determine the amount of cubic centimeters of material lost from the specimen, the following Material Volume Loss equation is determined based on ASTM D-5963-15:

$$MVL\ (\text{cm}^3) = \frac{(W1 - W2)\ \text{grams}}{\rho\ (\text{g}/\text{cm}^3)} \quad (6)$$

3 Results and Discussions

After conducting research on the production of rubber using three different types of filler materials, namely Zeosil (94% silica), Chemisil (98% silica), and Rice Husk Ash Silica (96% silica), the rheometer test results for the rubber components shown in Table 1, and the comparison graph can be seen in Figure 1.

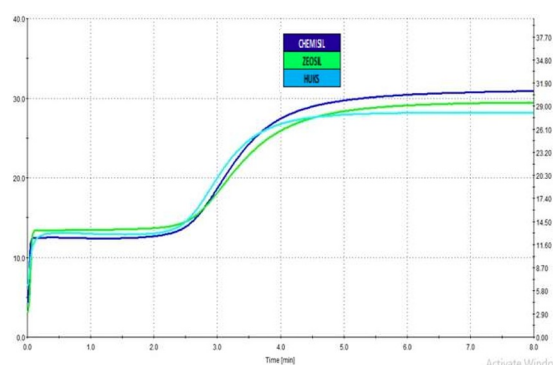


Fig. 1 The comparison of rheometer test result

Table 1 Rheometer test result

Test condition	Result	Standard	Chemisil	Zeosil	RHA
	MH	20 - 50 (dNM)	30.89	29.46	28.21
	ML	10 - 20 (dNM)	12.38	13.35	12.87
150°C × 480"	MH-ML	[dNM]	18.51	16.11	15.34
	T10	110- 165 (sec)	153.61	156.01	149.01
	T90	225-285 (sec)	272.67	279.87	257.72
	Status		PASS	PASS	PASS

The Rheometer was a compound testing process to determine the optimal temperature and time for

vulcanization. To obtain vulcanization results with optimal physical properties, the vulcanization duration of the rubber mixture must be determined precisely at a specific temperature t and the thickness of vulcanized rubber. Optimum maturity time (t_c 90) was the time required from the start of heating to mature the compound to optimum maturity. During the vulcanization process, the particle size of the filler used plays a very important role. The smaller the particle size, the easier the mixing process, so that the vulcanization process would be faster [10]. Figure 1 shows the variations in rheological behavior investigated during maturity time measurements. Scotch time (t_{c10}) and cure time (t_{c90}) of rubber composites filled with RHA and commercial silica (chemisil and zeosil) with RHA had shorter scotch time and cure time than chemisil and zeosil. RHA has less hydroxyl groups on its surfaces due to low specific surface area therefore cure retardation by the absorption of cures was reduced [11]. In addition, high metal oxides in RHA act as co-activators to reduce cure time. Similar results were found in RHA [12]. The addition of an activator led to a decrease in the scotch time and cure time of rubber composites. Silane coupling agent increased the amount of sulfur in the rubber composites that could cause crosslink reactions therefore scorch and cure time were reduced [11].

Maximum torque (MH) and minimum torque (ML) of Rubber composites are displayed in Table 1. The incorporation of filler into rubber increases MH and ML due to an increase in the stiffness of the composites and a reduction of the deformation of rubber molecules. Commercial silica showed higher MH and ML when compared to RHA. Silica had a smaller particle size than RHA resulting in better filler-rubber interaction [7]. The addition of an activator increased MH and ML of the composites.

Table 2 Mechanical properties of rubber composite

Properties	Unit	Chemisil (Silica 98%)	Zeosil (Silica 94%)	RHA (Silica 96%)
300% Modulus	kg/cm ²	35	37	42
Elongation length	%	768	830	867
Tensile	kg/cm ²	123	108	122
Hardness	Shore A	69	69	68
Tear	kg/cm ²	46	48	51
Abrasion	cc loss	0,19	0,12	0,17
Specific Gravity	g/cc	1,19	1,19	1,19

Rubber components with different filler contents were each tested for their mechanical properties using 300% Modulus, Abrasion, Hardness, Elongation, Specific gravity, Tear, and Tensile tests. Mechanical test results can be seen in Table 2. Tensile, elongation, and 300% modulus are affected by several factors, such as surface reactivity which determines the polymer-filler interaction, aggregate, size and shape of particles, structure, and filler particle dispersion in rubber [13].

Tensile strength is the amount of load required to stretch the test piece until it reaches the breaking point, measured in kilograms per cm² of cross-sectional area of the test piece before it stretched pro. An increase in the breaking stress value indicates that the elastic properties of the rubber component are increasing [14]. Elongation at break occurs because energy is used to stretch the bond between the vulcanizates molecules to break. Elongation at break is influenced not only by the ratio of natural rubber and synthetic rubber but also by the interaction of fillers and softening that form bonds between the molecular. Additives are added to natural rubber composites with synthetic rubber with the help of sulfur forming a cross-linked network [15].

The addition of active fillers and softeners to rubber composites can increase elongation at break, modulus, hardness, and tensile strength [16].

A Tensile Strength Tester is an instrument or tool used to perform tests to determine the properties of a component, such as flexibility (elongation), strength, 300% modulus, etc [13]. By pulling a material, we could quickly understand how the material reacts to tensile forces and determine the extent to which the element elongates.

Tensile Strength (Modulus) refers to how much load is required to stretch the test piece until it reaches a specific elongation, expressed per cm² of cross-sectional area of the test piece before stretching. Tensile stress is one of the physical properties of rubber materials whose main function involves size, structure, and the amount of filler added [17]. The tensile strength of rubber filled with chemisil and RHA is higher than zeosil. The incorporation of commercial silica chemisil and RHA improved the tensile strength of rubber composites compared to the tensile strength of zeosil composite due to the particle size of chemisil and RHA stood smaller than that of zeosil. Moreover, chemisil dan RHA contained a higher content of silica than RHA [12].

The value of 300% modulus was influenced by several factors such as surface reactivity which determines polymer-filler interactions, aggregates, particle size and shape, structure, and dispersion of filler articles in rubber. For RHA, the interaction between the filler and the hydrocarbon rubber was stronger than the attraction between the aggregates [13]. It was shown by the used rubber mixture and better rubber-filler interphase interaction, which were two important factors in determining the strength of the filled rubber-rubber mixture. The elastic properties of a finished rubber product were generated by the presence of cross-links between rubber particles. These cross-links occurred due to the vulcanization process. The compressibility of the

rubber was influenced by the temperature and duration of vulcanization, as the 50% compression set is influenced by the elasticity. Elasticity was affected by how complete the vulcanization process is [10].

Hardness refers to the resistance against indentation. The durometer is a tool that gauges how much a stress-loaded metal sphere penetrates the rubber. Rubber hardness was quantified in Shore A or Shore D units, following ASTM test procedures. Due to rubber's viscoelastic nature, a durometer reading peaks when the metal sphere achieves maximum penetration into the specimen and then diminishes in the next 5 to 15 seconds. While hand-held, spring-loaded durometers were widely used, they were prone to operator error. Bench-top dead-weight-loaded instruments minimize such errors [18]. The Hardness test was conducted to determine the hardness value of rubber vulcanizate under a specific compressive force [19].

The addition of filler materials tended to increase the hardness value of the final rubber product; the more filler added, the higher the hardness value. A lower hardness value in rubber tiles resulted in a softer texture (a more elastic surface). It was because the interaction between silica particles in the mixture enhances the stiffness of the compound. It could also be attributed to the ratio of the filler material, where rice husk ash (RHA) was more abundant than the total composition of the mixture. As a result, the interactions (Van der Waals forces and adsorption forces) between rubber particles and rice husk ash became imbalanced, with rice husk ash particles dominating, typically over the rubber particles [20]. Meanwhile, using smaller particle sizes promotes more uniform dispersion and homogeneity of rice husk ash particles in the rubber matrix, leading to better physical and mechanical strength properties of vulcanized natural rubber elastomer [21]. Hardness test results indicated that RHA has slightly lower hardness compared to chemisil and zeosil.

Tear resistance was the force required to pull apart a test specimen that has been punctured using a punch in the middle of the specimen along a 5 mm perpendicular direction to the pull force applied until rupture [14].

Determination of specific gravity is carried out to determine the quality of the rubber compound and calculate the amount of rubber needed for a certain volume. The principle of determining specific gravity is to weigh the sample in air and redo it in water. The weight of the test sample in water would be smaller than the weight of the sample in air because there was an upward pressure equal to the weight of the water being displaced, because the specific gravity of water is 1, the weight of the water being displaced was equal to the volume of the test sample.

The test results showed the same results for rubber components with chemisil, zeosil, and RHA fillers. It happened because the weight of the filler added for all types of filler was the same. The greater the addition of filler material, the more specific gravity increases. The smaller the particle size of the filler material, the more surface area would increase, so the specific gravity would decrease if the internal surface of the material was wider.

The increase in specific gravity is caused by more molecules in the silica compound (SiO_2) being bound to the rubber polymer, making the rubber compound denser so that the greater specific gravity [10].

The movement of bonds between molecules could lead to damage to the molecular structure of the material. The expansion or constriction of molecules during the aging process depended on the mechanical properties of the ingredients and the ratio of constituent materials. In this study, there were no differences in the specific gravity values. The specific gravity of the composites was primarily influenced by the density levels between the molecules of the material, although it was also affected by the compound milling process. The distribution of material within the rubber vulcanizate molecules, the vulcanization time, and the temperature changes during compound maturation also contribute to specific gravity variations [15].

The ability of softeners to stretch the molecular structure of the rubber composite during grinding influenced the speed at which additives and fillers enter the molecular structure. The distribution speed of the material into the rubber composite molecules during compound milling and the vulcanization reaction's speed, which forms a three-dimensional network, impact the density of cross-bonds and the overall density of the rubber composite. Moreover, differences in material ratios and types contribute to variations in specific gravity values. According to Hashim et al. [22], changes in specific gravity values were influenced by the ingredients used. An increase in the specific gravity of rubber can be attributed to an augmentation in cross-linking between rubber and filler material, leading to a reduction in the formation of pores [23]. The number of pores formed affected molecular density and specific gravity values.

The abrasion resistance was related to the rupture of tiny particles of the compound under the action of frictional forces, when sliding takes place between the compound surface and a substrate [4].

At particular loads, reinforcing fillers impart better abrasion resistance than inert fillers and ashes. The greater surface area and better rubber–filler interfacial adhesion could explain the obtained results. According to the reinforcement concept, the main factors governing elastomer's reinforcement, apart from filler loading, include filler-particle size or specific surface area, filler structure, and specific surface activity. Fine particles reflect their greater interface between the rubber matrix and the filler making it provide a higher degree of reinforcement than the coarse ones. The high-filler structure was always associated with processing difficulty as it increased considerably the viscosity of the compound and also the modulus of the vulcanizate. The surface activity was an important factor, indicating the extent of rubber–filler interaction. With good rubber–filler interaction, increased viscosity, modulus, and hardness, as well as other mechanical properties, will happen [9]. The mechanical properties results also show that the produced compound rubber could also be applied as sole rubber for shoes. This is due to the mechanical properties of rubber compounds complying with the industrial

standard for sole rubber, as shown in Table 3 [19]. In addition, the produced compound rubber also met the quality requirements for rubber components for vehicle window trim compound (SNI 06-1490-1989), dock bearing compound (SNI 06-3568-2006), SNI (06-7031-2004), handlebar grip compound motorized vehicles (SNI 06-7031-2004) [24].

Table 3 Standard for Nike Test Method

Property	Test Method	Standard		Unit
		Max	Min	
Tensile	Nike G76	100		kg/cm ²
Elongation	Nike G76	550		%
300% Modulus	Nike G76	35		kg/cm ²
Tear	ASTM D-624	35		kg/cm ²
Hardness	Nike G45	64	70	Shore A
Abrasion	Nike G9		0.35	Cc loss

4 Conclusions

The composites of rubber components has been successfully carried out using filler materials from commercial silica and rice husk ash (RHA). The results obtained from rheological tests indicate that RHA has a faster vulcanization time compared to chemisil and zeosil, with tc10 and tc90 values of 149.01 seconds and 257.72 seconds, respectively. Physical properties testing shows that RHA has better results in the 300% modulus, elongation, and tear tests compared to chemisil and zeosil. Meanwhile, for tensile strength, hardness, specific gravity, and abrasion resistance, RHA shows similar results and is not significantly different from chemisil and zeosil. From this research, it can be concluded that RHA can be used as an environmentally friendly filler material in the production of rubber components.

Based on the results of mechanical tests that had been done, the rubber compound obtained from this research met the requirements for the mechanical properties of rubber for shoe soles, vehicle window trim, dock bearings, motor vehicle handlebars and rubber compounds in general that were sold on the market.

Nomenclature

RHA Rice Husk Ash
 PHR Per Hundred Rubber

NR Natural Rubber
 IR Isoprene rubber
 KNB Acrylonitrile Butadiene Rubber
 TBzTD Tetrabenzyl thiuram disulfide
 MBTS Benzothiazole Disulfide
 EPR Ethylene Propylene Rubber
 MH Maximum torque
 ML Minimum torque

Greek Symbols

T_{max} T maximum torque
 T_i The torque at a specific curing time
 T_{min} The minimum torque
 L Maximum load (kg)
 t Thickness of the specimen (cm)
 w Width of specimen (cm)
 D_1 The original gauge length (cm)
 D_2 The elongated gauge length at the point of failure (cm)
 s The load when displaced by 10 cm
 t Thickness (cm)
 w The width of the material (cm)
 W_a The weight of the sample in air (g)
 W_w The weight of the sample in water (g)
 W_1 The weight of the material before testing (g)
 W_2 The weight of the material after testing (g)
 ρ The specific gravity (g/cm³)

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