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Araştırma Makalesi / Research Article

Characterization of Thermo-Physical Properties of Nanoparticle Reinforced the Polyester Nanocomposite

Nanopartikül Takviyeli Polyester Nanokompozitin Termo-Fiziksel Özelliklerinin Karakterizasyonu

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ABSTRACT

In this research, some thermal and physical properties of unsaturated polyester (UP) nanocomposite produced using nanoparticles were investigated. Fumed Silica (FS), silicon carbide (SiC) and graphene nanoplate (GNP) were used as nanoparticles. The synthesized polyester nanocomposites were reinforced with 0.2%, 0.4%, 0.6%, 0.8% and 1.0% nanoparticles by mass. GNP and SiC nanoparticle reinforcements increased the thermal conductivity coefficient of the produced polyester nanocomposite by approximately 64% and 39%, respectively, while FS reinforcement decreased it by 12.5%. SiC nanoparticle increased the Shore D hardness of the polyester nanocomposite by 5.26% in the sample with 0.1% reinforcement. This ratio was 3.85% for 0.1% supplemented FS and 1.9% for 0.4% GNP. In addition, SiC and GNP reinforcement increased the density of the polyester nanocomposite, while FS reinforcement decreased the density of the composite. The thermal stability order of the samples was determined as FS, GNP and SiC reinforced nanocomposites. It was determined that the sample with the lowest thermal stability was UP. According to the results obtained in thermal stability experiments, mass losses of reinforced nanocomposites during thermal decomposition were compared. The thermal decomposition behavior of polyester nanocomposites was modeled by the kinetic equation. Experimental and theoretical model results were compared and correlation numbers were calculated by statistical analysis using nonlinear regression.

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ÖZ

Bu araştırmada, nanopartiküller kullanılarak üretilen doymamış polyester (UP) nanokompozitinin bazı termal ve fiziksel özellikleri araştırılmıştır. Nanopartikül olarak Füme Silika (FS), silisyum karbür (SiC) ve grafen nanoplate (GNP) kullanılmıştır. Sentezlenen polyester nanokompozitlere

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kütlece %0.2, %0.4, %0.6, %0.8 ve %1.0 oranlarında nanopartikül takviye edilmiştir. Üretilen polyester nanokompozitin ısıl iletkenlik katsayısını GNP ve SiC nanopartikül takviyeleri sırasıyla yaklaşık %64 ve %39 oranında arttırırken, FS takviyesi %12.5 oranında azaltmıştır. SiC nanopartikül, polyester nanokompozitin Shore D sertliğini % 0.1 takviye edildiği numunede %5.26 oranında arttırmıştır. Bu oran % 0.1 takviyeli FS için %3.85 ve %0.4 GNP için %1.9 olmuştur. Ayrıca, SiC ve GNP takviyesi polyester nanokompozitin yoğunluğunu arttırırken, FS takviyesi kompozitin yoğunluğunu azaltmıştır. Numunelerin termal kararlılık sıralaması FS, GNP ve SiC takviyeli nanokompozitler olarak tespit edilmiştir. En düşük termal kararlılığa sahip numunenin UP olduğu belirlenmiştir. Termal kararlılık deneylerinde elde edilen sonuçlara göre, güçlendirilmiş nanokompozitlerin termal bozunma sırasındaki kütle kayıpları karşılaştırılmıştır. Polyester nanokompozitlerin termal bozunma davranışı kinetik denklem ile modellenmiştir. Deneysel ve teorik model sonuçları karşılaştırıldı ve doğrusal olmayan regresyon kullanılarak istatistiksel analiz ile korelasyon sayıları hesaplanmıştır.

1. INTRODUCTION

Polymeric composites made with unsaturated polyesters have become very common and popular in recent years. Such composites, which can be applied in the laboratory environment and are easier to process mold, and cure, can be given different properties suitable for them. Especially with nanoparticles, polyester composites can be developed according to the purpose of use.

Many studies have been carried out in the literature to increase the thermal stability of polyester composites. Inorganic additives such as ammonium polyphosphate impart non-flammability to unsaturated polyester and increase its thermal stability [1-5]. When expandable graphite, silica, melamine, some phosphorus, and halogen compounds are used as fillers, both thermal and mechanical properties of polyester have been improved [6-8]. Also, the use of metal particles such as aluminum, copper, zinc, stainless steel, silver, gold, and nickel in the polymer matrix gives the polyester composite flame retardant properties [9, 10].

Physical decomposition in composites obtained from unsaturated polyester continues up to 120 °C. If there are hydrated compounds in the composite structure, the removal of hydrated water can reach 160 °C. At higher temperatures, volatile chemical compounds may be removed from the composite structure. Chemically, the thermal decomposition phase starts after a temperature of about 260 °C and occurs very quickly. Inorganic impurities remaining in the polyester composite without decomposition can remain in the composition of the waste ash above 700 °C [11-13].

Nanoparticles have started to be used thanks to the technological developments that have developed in addition to micro-structure additives and fillers. It has been determined that nanoparticles such as carbon nanotubes and graphene strengthen the mechanical and physical properties of the polyester composite. In the researches, carbon nanotube and graphene reinforcement gave good results in mechanical tests of the polyester nanocomposite, even at low mass ratios. Also, nanoparticles such as nano-silica and glass fiber improve the mechanical properties of the polyester composite [14-16].

In other studies, on unsaturated polyester, thermal decomposition kinetics of the reinforced composites have been investigated. Using the model equations in the literature, results can be evaluated with thermogravimetric analysis for the polyester composites [17-19].

When the studies in the literature are examined, although there are studies in which GNP, SiC and FS nanoparticles are used separately, there is no study in which all three are used together. In this study, GNP, SiC, and FS were used as reinforced nanoparticles in unsaturated polyester. The effects of these nanoparticles on the thermal conductivity coefficient, Shore D hardness, density, and thermal stability of the produced composite have been evaluated. The reinforcement of these nano-sized particles can improve some desired properties of the polyester composite.

2. MATERIAL AND METHOD

2.1 Materials

Orthophthalic based unsaturated polyester resin (UP), methyl ethyl ketone peroxide (MEKP), and cobalt octoate (Co. Oc.) used in this study were supplied from Turkuaz Company. Graphene (GNP) is 99.9% pure, 3nm thick, 1.5 µm in diameter, 2267 kg/m3 density, and has a surface area of 800 m2/g. Fumed Silica (Aerosil 300) has a purity of 99.8%, a surface area of 300 m2/g, a tamped density of 55 kg/m3, and a particle diameter of 7 nm. Silicon Carbide (SiC) has 99.5% purity, 50-70 nm size, 3320 kg/m3 density, and 40-85 m2/g surface area. All nanoparticles have been supplied from Nanography.

2.2 Methods

Table 1 shows the utilization rates of nanoparticles in the polyester nanocomposite, and Figure 1 shows the production scheme of the nanocomposite obtained with synthesized unsaturated polyester. Free radical decomposition can occur rapidly by cobalt ions by redox reactions at low temperatures, and free radicals can be formed.

$$ROOH + Co^{2+}L_2 \xrightarrow{k_1} RO^{\bullet} + OH^{-} + Co^{3+}L_3 \quad (1)$$

$$ROOH + Co^{3+}L_3 \xrightarrow{k_2} [ROOHCo^{3+}L_3] \xrightarrow{k_3} ROO^{\bullet} + H^{+} + Co^{2+}L_2 \quad (2)$$

Free radicals (RO● and ROO●) and octoate compound (L) are seen in chemical reactions (1) and (2) [20].

Table 1. Experimental study plan of nanoparticles

Sample No	Nanoparticle Ratio (wt.%)	UP (wt.%)	MEKP (wt.%)	Co. Oc. (wt.%)
1	0.0	98.4	1.2	0.4
2	0.2	98.2	1.2	0.4
3	0.4	98.0	1.2	0.4
4	0.6	97.8	1.2	0.4
5	0.8	97.6	1.2	0.4
6	1.0	97.4	1.2	0.4

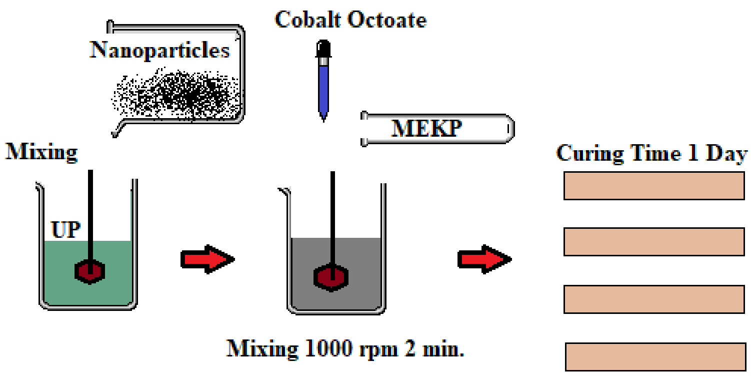

Figure 1. *Experimental working scheme of the produced polyester nanocomposite*

2.3 Thermal Decomposition Kinetic

In the equations below, the conversion rate (α) is expressed as initial mass (Mi), final residual mass (Mf), and time-varying mass (Mt). The thermal degradation of polyester nanocomposite has been investigated by chemical reaction kinetics. Here, reaction rate constants (k1, and k2), degree of chemical reaction (m, and n), rate of temperature increase (β), and conversion function (f(α)) are used in the equations. The change of conversion with time in a chemical reaction is shown in Eq. 2, and the change of conversion with temperature is shown in Eq. 5. The thermal degradation kinetics of the produced polyester nanocomposites have been modeled according to Equation 6 by performing the necessary integration processes after the equations are arranged. The model equation of the conversion rate (α) obtained by making the necessary simplifications, depending on the temperature (T), K, k_1, and k_2 are given in Eq. 7.

$$\alpha = \frac{M_t - M_f}{M_i - M_f} \quad (1)$$

$$\frac{d\alpha}{dt} = k(T) \cdot f(\alpha) \quad (2)$$

$$f(\alpha) = (1-\alpha)^n \quad (3)$$

$$k(T) = (k_1 + k_2 \cdot T)^m \quad (4)$$

$$\frac{d\alpha}{dT} = \frac{1}{\beta}((k_1 + k_2 \cdot T)^m)(1-\alpha)^n \quad (5)$$

$$\frac{(1-\alpha)^{1-n}}{(n-1)} = \frac{(k_1 + T \cdot k_2)^{m+1}}{\beta \cdot (m+1) \cdot k_2} \quad (6)$$

$$\alpha = 1 - [\frac{(k_1 + T \cdot k_2)^{\frac{(m+1)}{(1-n)}}}{K}] \quad (7)$$

The thermal decomposition behavior of the polyester nanocomposite has been re-improved with the help of kinetic equations in the literature [21-30].

3. RESULTS AND DISCUSSIONS

When the results obtained in the experimental studies have been evaluated, it is determined that the thermal conductivity coefficient of the polyester nanocomposite rose as the GNP ratio increased. When Figure 2 has been examined in detail, it is seen that the thermal conductivity coefficient of the obtained polyester nanocomposite is increased by GNP and SiC nanoparticles, while FS decreased it. In other words, FS nanoparticles improved the insulation property of the produced polyester nanocomposite.

In Figure 3, although the Shore D hardness of GNP reinforced polyester nanocomposite tends to increase, it does not change much at high rates. It is seen that the Shore D hardness increases as the SiC nanoparticle ratio (wt%) rise in the polyester nanocomposite. It has been determined that polyester nanocomposite FS reinforcement also increased Shore D hardness.

In Figure 4, the effect of the nanoparticle ratio in the polyester on the density of the nanocomposite has been evaluated. Although the density of polyester nanocomposite reinforced with SiC and GNP nanoparticles increased, the addition of FS filler decreased the density of the composite.

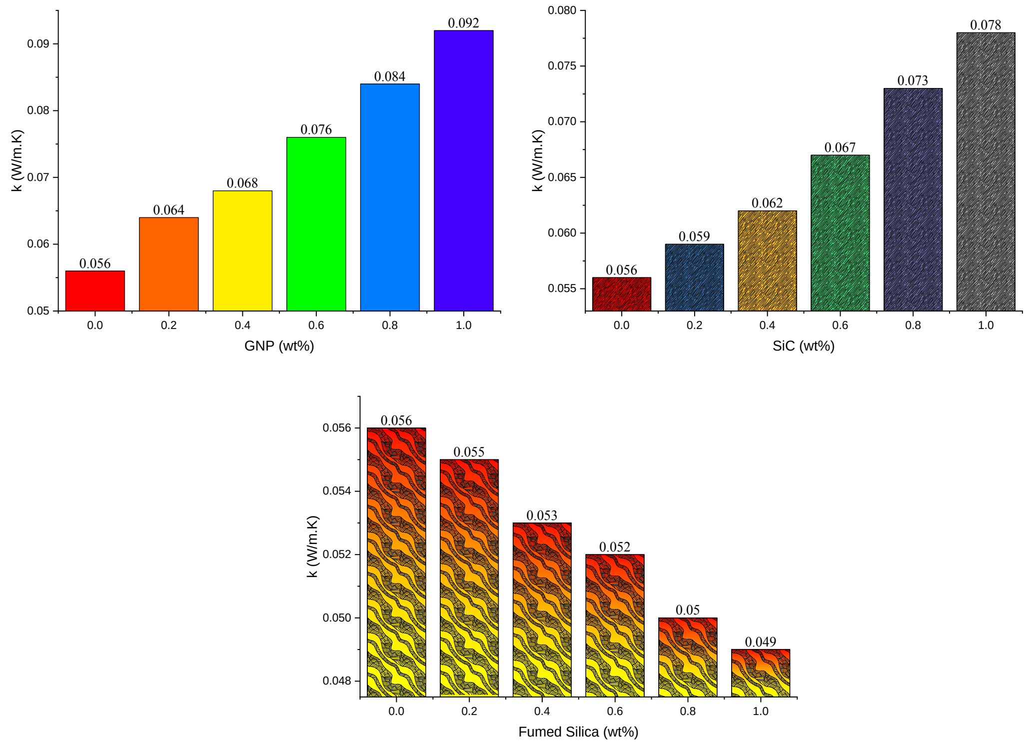

Figure 2. *Effect of GNP, SiC, and FS reinforcement on thermal conductivity of the polyester nanocomposite*

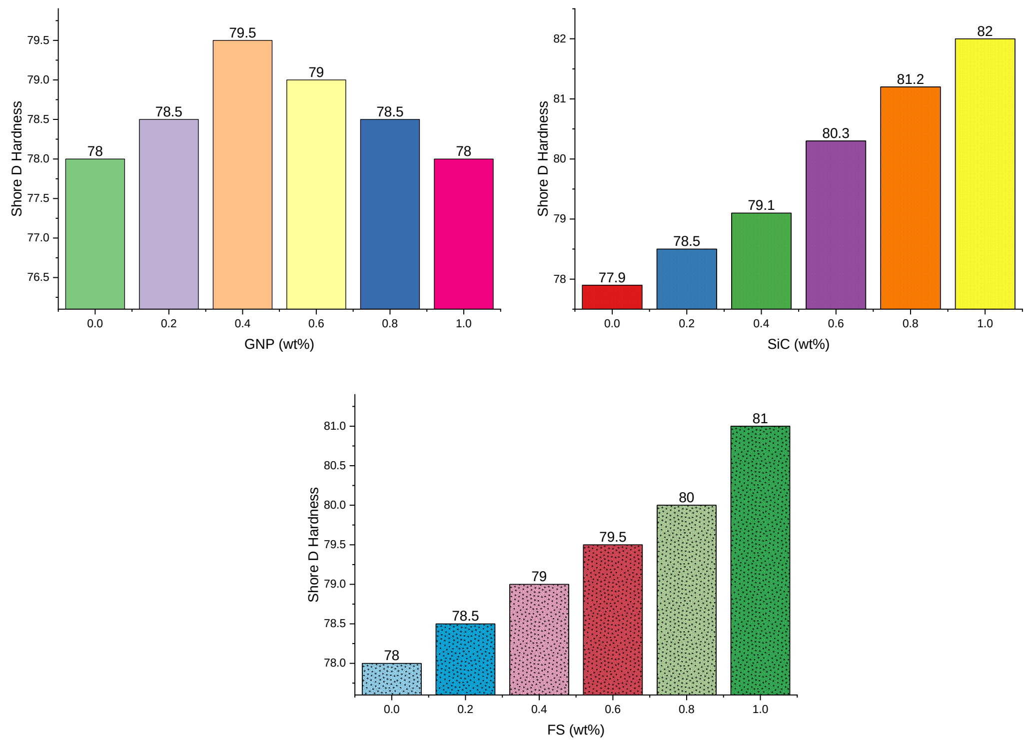

Figure 3. *Effect of GNP, SiC, and FS reinforcement on Shore D hardness of the polyester nanocomposite*

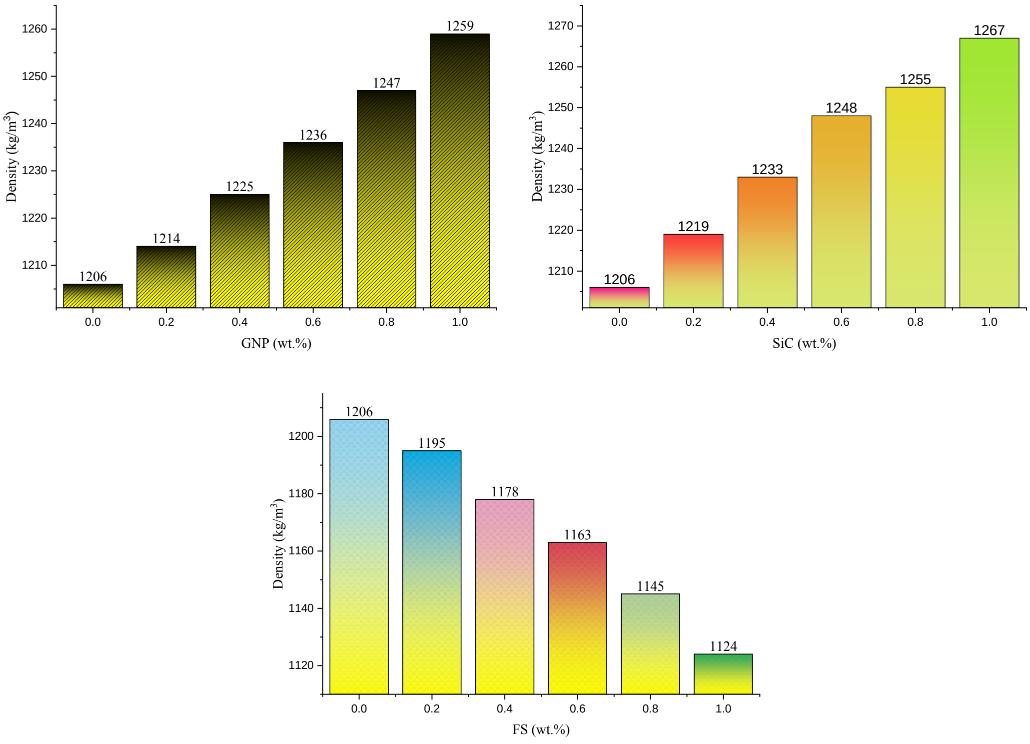

Figure 4. *Effect of GNP, SiC, and FS reinforcement on the density of the polyester nanocomposite*

In Figure 5, the thermal degradation behavior of the nanoparticles has been compared with the temperature increase. The fastest thermal decomposition is observed in pure unsaturated polyester, and the slowest thermal degradation is seen with 1 wt% FS reinforced polyester nanocomposite. The thermal stability of 1% GNP reinforced polyester nanocomposite is found to be better than 1% SiC reinforced nanocomposite.

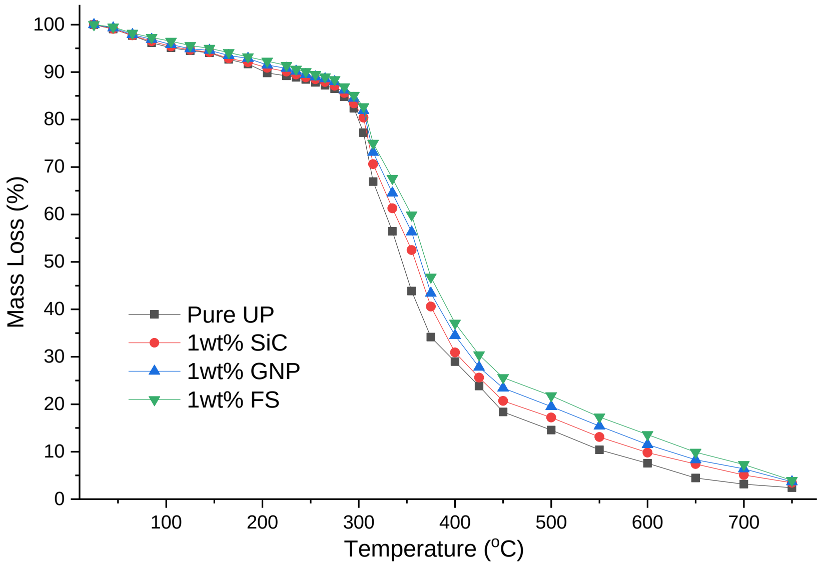

Figure 5. *Effect of GNP, SiC, and FS reinforcement on thermal decomposition of the polyester nanocomposite*

Thermal decomposition experiments of the polyester nanocomposite have been carried out in the PID-controlled system with a temperature increase of 10 K/min. The thermal degradation behavior of polyester nanocomposite has been evaluated with conventional thermogravimetric analysis. The mass losses of polyester nanocomposites shown in Figure 5 are applied to the kinetic model in Equation 7. The correlation coefficients calculated with the help of statistical analyzes are given in Table 2. In Table 3, the calculated root mean square error (RMSE), a sum of squares total (SST), chi-square (χ^2), and correlation coefficients (R^2) values are expressed. The compatibility between the experimental data and the theoretical model has been determined by the minimum error functions and the maximum R^2 values.

Table 2. Coefficients of the kinetic model found in Equation 7

Experiments	k_1	k_2 (1/ºC)	m	n	K
Pure UP	-1.51911	0.00519	-0.61686	0.02694	1.17309
1 wt% SiC	-6.39970	0.02136	-0.57548	0.60183	5.32471
1 wt% GNP	-4.85423	0.01625	-0.53986	0.62924	5.45855
1 wt% FS	-7.15376	0.02409	-0.49685	0.60897	6.82943

Table 3. Evaluation of the kinetic model with statistical analysis

Experiments	RMSE	SST	R^2	χ^2
Pure UP	0.03802	0.64332	0.97581	0.08265
1 wt% SiC	0.06033	0.72420	0.96298	0.08160
1 wt% GNP	0.01521	0.58075	0.98114	0.07374
1 wt% FS	0.02901	0.59884	0.97760	0.08027

4. CONCLUSION

When the experimental results of the produced polyester nanocomposites have been evaluated:

(i) FS reinforced composite had the highest thermal stability, while pure polyester nanocomposite (UP) had the lowest.

(ii) The lowest density and thermal conductivity coefficient have been obtained in FS added polyester nanocomposite [31-33]. The thermal conductivity coefficient of polyester nanocomposite was lowered by 12.5% due to FS reinforcement.

(iii) With SiC filler, polyester nanocomposite has got maximum Shore D hardness [34-36]. SiC nanoparticles increased the Shore D hardness of the polyester nanocomposite by 5.26% in the sample with 0.1% reinforcement. This ratio was 3.85% for 0.1% supplemented FS and 1.9% for 0.4% GNP.

(v) Obtained with the highest density polyester nanocomposite SiC reinforcement.

(iv) Graphene nanoparticle (GNP) increased the thermal conductivity coefficient of polyester nanocomposite the most. The thermal conductivity coefficient of the generated polyester nanocomposite was improved by roughly 64% and 39%, respectively, with the addition of GNP and SiC nanoparticle reinforcements.

Hence, FS nanoparticles should be preferred to produce a composite with low density, good insulating properties, and high thermal stability. SiC nanoparticles may be used to obtain polyester nanocomposites with high density and hardness. GNP nanoparticles can be preferred to synthesize polyester nanocomposites with high thermal conductivity.

CONFLICTS OF INTEREST

No conflict of interest was declared by the authors.

AUTHORS' CONTRIBUTIONS

Cenk YANEN: Visualization, methodology, validation, analysis, data collection, writing-review and editing, supervision. Ercan AYDOĞMUŞ: Writing-original draft preparation, analysis, data collection, data curation, conceptualization.

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