

High Quality Fused Silica from Egyptian Silica Sand Concentrate

T. R. Boulos¹, Ahmed Yehia², Mohamed B. Morsi³, S. S. Ibrahim⁴
^{1,2,3,4}Central Metallurgical R&D Institute, P.O.Box 87, Helwan, Cairo, Egypt
(¹trboulos@yahoo.com)

Abstract- White silica sand from the Eastern Desert of Egypt was subjected to intensive beneficiation program to match the international grade requirements of ultra-pure fused product. Size distribution analysis of the sand sample indicates that by rejecting the +0.4 mm coarse fraction, representing about 24.7% by weight and assaying 0.036% Fe₂O₃, the -0.4 mm undersize assays only 0.013% Fe₂O₃. Attrition scrubbing of this size cut followed by a combined flotation-magnetic separation circuit, final sand concentrates assaying 0.0059% Fe₂O₃ and 0.015% Al₂O₃ was obtained with an overall recovery of 70%. Chemical leaching of this concentrate, by using HCl and stannous chloride at 50-60°C, led to the production of ultra-pure sand concentrate having 0.0043% Fe₂O₃. Fused silica was prepared using either an arc furnace or an electrical resistance heating Tamman furnace. The arc furnace product was greyish brown in colour and coated with carbon dissipated from the graphite hearth and the electrodes which affected its chemical constitution and physical properties. But product of the electrical Tamman furnace was clear and exhibited excellent physico-mechanical properties that satisfy very well the requirements for high temperature refractory materials, special refractory industries, investment casting and as a filler for epoxy molded resins.

Keywords- *Fused Silica Sand, Flotation, Magnetic Separation, Attrition Scrubbing*

I. INTRODUCTION

Silica sands, perhaps, have got the most diversified use among all the non-metallic minerals. This is because of its common occurrence around the world, and due to distinctive physical characteristics such as hardness, chemical and heat resistance as well as low price. Silica bearing rocks and minerals such as quartz, quartzite, silica sand together with other varieties of silica like agate, amethyst, jasper, flint etc. are used in a host of industries such as glass, ceramics, foundries, ferro-alloys, abrasives, refractories, ornamentation etc. The uses of silica sand depend on its mineralogy, chemistry and physical properties. Impurities usually present in the silica sand are free and coated iron oxides, clay, titania and smaller amounts of sodium, potassium and calcium minerals.

Upgrading of silica sand requires partial removal of iron, and other minerals which are detrimental to its end use, [1-3]. Much of the liberated impurities can be reduced or removed by physical operations such as size separation (screening), gravity separation (spiral concentration), magnetic separation etc., [4-7] Sometimes, physico-chemical (flotation) or even chemical methods (leaching etc.) are to be adopted for effective removal of iron oxides which may be in intimate association with the mineral quite often superficially, [8-11]. The most appropriate method depends on the mineralogical forms and distribution of iron in the ore, [12, 13]. To increase the value of such sand concentrate, it is possible to use it for the production of expensive fused silica, [14-16]. Fused silica is produced by fusing (melting) high-purity silica sand (>98% SiO₂) at approximately 1650 °C, [17]. This could be successfully accomplished by one of four alternative methods: carbon electrode, carbon arc, plasma arc or gas fired continual extrusion [18, 19]. Carbon electrode technology represents the bulk of world production, although alternative technologies are gaining wider acceptance due to their capacity to produce high quality fused silica. The carbon electrode technology is relatively straightforward. However; the production of fused silica represents a significant level of value addition from a low cost starting material. Fused silica is an excellent refractory with a very high resistance to thermal expansion and acid attack. It is particularly useful where extremes of temperature occur. Most of the applications of fused silica exploit its wide transparency range, which extends from the UV to the near IR, [24-26]. Fused silica is the key starting material for optical fiber, used for telecommunications. Because of its strength and high melting point (compared to ordinary glass), fused silica is used as an envelope for halogen lamps and high-intensity discharge lamps, which must operate at a high envelope temperature to achieve their combination of high brightness and long life. Vacuum tubes with silica envelopes allowed for radiation cooling by incandescent anodes. The dielectric properties and very high electrical receptivity of these materials over a wide range of temperatures together with their low thermal conductivity allow their use as an electrical and thermal insulating material in wide range of environments. The combination of thermal, chemical and UV stability together with high DUV transparency makes them an excellent choice for projection masks in photolithography.

II. MATERIALS AND EXPERIMENTAL TECHNIQUE

A. Materials

A three tone sample representing El-Zaafarana locality was supplied to CMRDI by Nile Mining Co. Yard sampling of the sand was carried out by coning and quartering methods to

prepare about 5 kg batches. One of these batches was used in particle size distribution measurements and complete chemical analysis. Complete chemical analysis of the raw sand, as carried out by XRF methods, shows that it is of good quality with 99.32% SiO₂, 0.02% Fe₂O₃ and 0.04% Al₂O₃, Table 1.

TABLE I. COMPLETE CHEMICAL ANALYSIS OF ZAAFARANA SAND SAMPLE

Constituent	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	Cl	SO ₃	L.O.I.
%	99.32	0.03	0.04	0.02	0.01	0.02	0.06	< 0.01	< 0.01	0.01	< 0.01	< 0.01	0.3

B. Experimental technique

1) The attrition scrubbing process

This was carried out in the laboratory using a 'Denver D12' flotation cell conditioner at pre-determined optimum conditions of 65% solid, impeller speed of 2500 rpm and at 40 min. This was done to get rid of the ferruginous clayey coating of the silica grains. Classification of the attritioned product was carried out on 0.1 mm screen, the oversize of which was collected, dried and chemically analyzed for Fe₂O₃ and Al₂O₃.

2) The different beneficiation options of the attritioned sand

Anionic flotation of the heavy mineral impurities was, primarily, conducted using the 'Denver D12' flotation cell, which is a simulation of the subaeration type, with 3 kg/ton of the local dodecyl benzene sulphonate collector at pH 3. In the meantime, the 'Eriez' high intensity magnetic separator was used as an alternative to flotation to pick up the entrained magnetic minerals from the classified sand. This process was also combined with the flotation process looking for ultrapure silica concentrate. Chemical leaching of the silica concentrate was conducted using HCl and stannous chloride (as a reducing agent) at temperature 50-60°C for 2 hrs. The final silica concentrate was collected, dried and analyzed.

3) The Laboratory Carbon Arc Fusion

A submerged baby electric arc furnace of 25 kW was employed on bench scale in the fusion of the white sand concentrate, Fig. 1, [20, 21]. The furnace, of about 2 kg capacity, is composed of a conical shaped graphite hearth 5 cm diameter at the bottom, 10 cm at the top and 20 cm height. The hearth with a 20 mm graphite electrode at its center was used for producing the spark and melting the charge. Another graphite electrode attached to the bottom of the hearth and ending with a copper joint complements the fusion circuit. A refractory lined steel shell surrounding the hearth keeps the temperature of the charge during the fusion process. The furnace is furnished with a variable AC transformer for power supply and a device for horizontal tilting for charge disposal. About 500-1000 g samples of crude silica sand and its concentrate were charged into the furnace with a rate of 50 g/min. The process lasts 2-3 hours from charging the furnace to cast the fused product. After complete melting of all charge it was hold in the furnace, under controlled temperature of 1850 °C for silica sand for a time varying between 10 and 45 min.

The fused sample was, then, casted in a graphite mold and left to cool down. After cooling, it was subjected to jaw crushing to -150 mesh screen and kept in plastic bags for further physicochemical evaluation. Crude silica sand was primarily used in the fusion experiment before shifting to its concentrate to demonstrate the effect of metal impurities on the quality of the product.



Figure 1. Photograph of the Baby Electric Arc Furnace used for preliminary fusion tests

4) The laboratory Tamman furnace

A laboratory Tamman furnace, Fig.2, was used for electrical fusion of crude silica sand and its concentrate, as an alternative to the arc furnace fusion process, [22, 23]. This was decided in view of the dark grey colour of the fused products of this latter furnace due to contamination with graphite dust. The Tamman furnace does not cause such a problem. It is mainly composed of a graphite crucible 20 cm length and 2.5 cm diameter. The crucible is heated inductively using three concentric graphite heating elements separated by graphite

pieces 1-3 mm in size. The crucible is covered by two insulating water-cooled steel covers to maintain the furnace temperature and minimize heat dissipation. Two copper electrodes are connected to the heating elements to transfer the electric current coming from a variable transformer to the furnace. Stepwise increase in the furnace temperature by increasing its amperage to 150 A and the voltage to 15 V was conducted every experiment until it reaches the desired temperature at 1850 °C for silica sand.



Figure 2. The Tamman Furnace used in production of fused silica

About 100 g samples of the white sand concentrate were used in the fusion experiments. After charging the furnace with the raw materials it was heated gradually to the desired temperature. The furnace temperature was controlled between 1780°C and 2050°C. After complete melting of all the charge which normally takes time of 3-4 hrs, it was then, hold in the furnace for a certain time that varied from 15-45 min. At the end of the holding time of the sample, the fused product was casted in a suitable graphite mold and left to cool down. After cooling the fused product, it was crushed to pass 150 mesh sieve and kept for physico-chemical evaluation in a plastic bag. The powder sample was annealed at 1000 °C for 2 hrs in a muffle furnace to remove any residual carbon from the sample surface.

III. RESULTS AND DISCUSSION

A. Sample characterization

The fraction above 400µm, which is about 24.3 % of the feed sample, is the coarse grits and is taken separately since it is not suitable for glass making. Similarly, the fraction below 180µm (~0.82% of feed sample) which contains mostly kaolin (clay mineral) is also rejected since the same is also unsuitable for glass making. The weight fraction of -400 +180µm 'cut' is about 74.88%, which, as per the size specification, is the 'desired fraction for the white silica sand'. However, size distribution analysis indicates that by rejecting the + 0.4 mm

coarse sand, representing about 24.3 % by weight and assaying 0.036% Fe₂O₃, the -0.4 mm fraction reaches 0.013 % Fe₂O₃, Table 2.

TABLE II. DRY SIZE DISTRIBUTION ANALYSIS OF EL-ZAAFARANA WHITE SAND SAMPLE

Size, mm	Wt %	Cum.Wt%. Retained	Fe ₂ O ₃ %
+0.6	10.74	10.74	0.037
-0.6+0.42	13.56	24.30	0.035
-0.42+0.21	56.90	81.20	0.013
-0.21+0.18	17.98	99.18	0.0139
-0.18	0.82	100.00	0.168
Total	100		0.0199
Head			0.020

B. The Attrition Scrubbing Process

Since the combined iron was found to be mainly as coating on the surface of the sand grains, it was decided to go for attrition followed by flotation and magnetic separation.

The scrubbed sand was then classified by screening on 0.1 mm screen. This technique proved to be very effective worldwide in disintegrating the ferruginous clayey coating of the sand. Table 3 shows that 40 min attritioning time, of the classified sand sample, are enough to get an iron oxide value of 0.01% and 0.02% Al₂O₃. Though the impurity level has been reduced, the silica content is still below the required level and the iron value is more than the specified value.

TABLE III. EFFECT OF ATTRITIONING TIME ON THE CLEANING OF THE CLASSIFIED SAND SAMPLE

Attritioning Time, min.	10	20	30	40
Fe ₂ O ₃ %	0.0109	0.0106	0.0103	0.0100
Al ₂ O ₃ %	0.029	0.026	0.022	0.020

C. Flotation of the Attritioned Sand

By using 3kg/t of the local dodecyl benzene sulphonate as a collector for the heavy minerals, at pH 3, a sand concentrate assaying 0.0087% Fe₂O₃ and 0.018 %Al₂O₃ was obtained with a recovery of 96%.

D. High intensity magnetic separation of the attritioned sand

The magnetic separation, which is integral with the attrition, was carried out using the most advanced "Eriez" rare earth, RE; magnetic roll which was operated on batch basis. Process optimization included verification of the feed rate and number of cleaning steps. Results indicate that with such belt separators, the grade of the concentrate improves by decreasing the feed rate. However, by increasing the number of cleaning operations, the grade of the sand concentrate reaches 0.0088% Fe₂O₃ and 0.019% Al₂O₃ after 3 separation steps and goes down to 0.0080% Fe₂O₃ and 0.017% Al₂O₃ after 4 steps.

E. The combined sand flotation – magnetic separation circuit

When the attritioned sand was subjected to a combined flotation – magnetic separation circuit, under the predetermined optimum conditions, a final concentrate assaying 0.0059% Fe₂O₃ and 0.015 % Al₂O₃ was obtained with an overall weight recovery of 70%. Evidently both processes complement each other for maximum removal of iron oxide impurities.

F. Chemical leaching of the sand concentrate

Oxalic acid has been recommended worldwide as an environmentally friendly reagent for dissolving iron oxide from sand surface, [4-10]. It was found to react with Fe (III) ions forming soluble complexes depending on the iron minimal concentration. The process proved to complement other

physical separation methods in the production of high quality silica products. Experimental results showed that the application of concentrated HCl and stannous chloride as reducing agent was more efficient to remove the remaining iron oxide on the silica grain surface than using of oxalic acid. Chemical leaching of the final sand concentrate, assaying 0.0059% Fe₂O₃, was conducted using the "D12" flotation conditioner at high speed (2500 rpm) and by the addition of 4 kg/t HCl and 0.4 kg/t stannous chloride at temperature of 50-60°C, [27]. The leaching process was applied for 2 hrs producing a concentrate assaying 0.0043% Fe₂O₃. The overall operational laboratory beneficiation tests of the classified sand sample, -0.40+0.18 mm, are shown in Table 4. Complete chemical analysis of the sand concentrate is shown in table 5.

TABLE IV. OPERATIONAL LABORATORY BENEFICIATION TESTS OF THE WASHED SAND SAMPLE

Product	Washed Sample	Washed flotation concentrate	Washed non-magnetic concentrate	Washed flotation non-magnetic concentrate	Chemically leached final product
Operational Wt%	99.00	96.00	74.00	70.00	70.00
Fe ₂ O ₃ %	0.010	0.0087	0.0080	0.0059	0.0043

TABLE V. COMPLETE CHEMICAL ANALYSIS OF THE SAND CONCENTRATE SAMPLE

Constituent	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Cl	Na ₂ O	CaO	K ₂ O	P ₂ O ₅	TiO ₂	MgO	SO ₃	MnO	L.O.I.
%	99.96	0.013	0.0043	0.00	0.004	0.007	0.003	0.001	0.00	0.0009	0.0009	0.00	0.00

TABLE VI. SIZE DISTRIBUTION ANALYSIS OF THE WHITE SAND CONCENTRATE

Size, mm	Sand Concentrate	
	% Wt.	% Cum. Weight Retained
-0.6+0.41	5.46	5.46
-0.41+0.21	80.40	85.86
-0.21+0.18	14.14	100

G. Fused silica production from the silica sand concentrate

1) Fused silica production using electric arc furnace

The purity of the silica input, as previously reported is very crucial in the production process of fused silica. Unlike steelmaking and similar metal production processes, the electrofusion process has no slag phase to remove impurities. As a result, all deleterious materials that enter the electrofusion process via the raw materials are retained within the end product, significantly, affecting its grade and performance characteristics. In this respect, complete chemical analyses of the white sand concentrate employed in the fusion process are shown in Table 5 and their grain size distribution analyses are depicted in Table 6. With 99.96% silica content and total impurities less than 50 ppm the raw materials comply very well with quality standards of the electrofusion process. In terms of

particle size, this parameter will be optimized in the small scale production process. Crude silica sand was primarily used in the fusion experiments, using electric arc furnace before shifting to its concentrate to demonstrate the effect of metal impurities on the quality of the product. The fused silica product from crude silica sand was dark in color due to carbon contamination from the fusion process. Complete chemical analysis of the produced fused silica showed that it contains about 0.42% C which results in a similar decrease in its SiO₂ content, Table 7. Ore microscopy, on the other hand, showed dark spots of carbon in the yellowish brown fused silica, sometimes forming carbides or including metal oxides of the silica sand impurities, Fig. 3. It was stated that in the furnace configuration, the fusion process and the quality of the starting raw materials dictate the purity of the fused silica end product.

TABLE VII. COMPLETE CHEMICAL ANALYSIS OF FUSED SILICA PRODUCED BY THE ARC FURN

Constituent	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Na ₂ O	CaO	K ₂ O	P ₂ O ₅	TiO ₂	MgO	MnO	L.O.I.	C
%	99.52	0.025	0.003	0.004	0.008	0.003	0.002	0.005	0.001	0.008	0.00	0.42



Figure 3. Photomicrograph of fused silica product from Electric Arc Furnace showing flowage texture staining with iron oxide. Plain polarized light

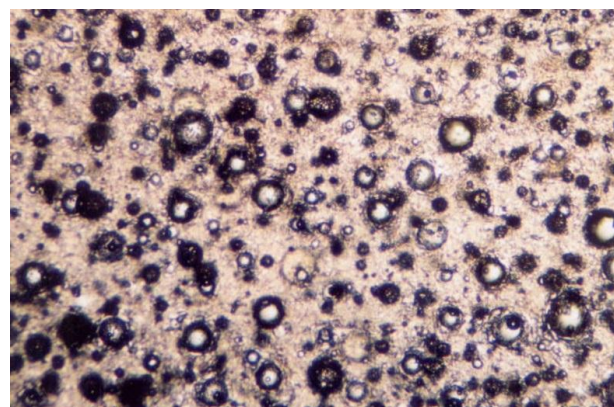


Figure 4. Photomicrograph of fused silica sample produced from the Tamman Furnace at optimum fusion condition showing carbon layer surrounding the grains of the fused silica

2) The Tamman Electrical Fusion Process

A laboratory Tamman furnace was used for electrical fusion of white sand concentrate as an alternative to the arc furnace fusion process. This was decided in view of the dark grey colour of the fused products of this latter furnace due to contamination with graphite dust. The Tamman furnace does not cause such a problem. Process optimization for the silica sand sample included verification of the fusion temperature, feed particle size, feeding rate and holding time after melting in the heating zone, Table 8, Fig.4. Annealing of the fused product was employed as a measure for getting rid of the carbon surface coating of the silica melt, which after burning leaves the fused product surface clean, Fig. 5. However, the process gave much better results with the Tamman furnace fusion products as compared with that of the arc furnace.

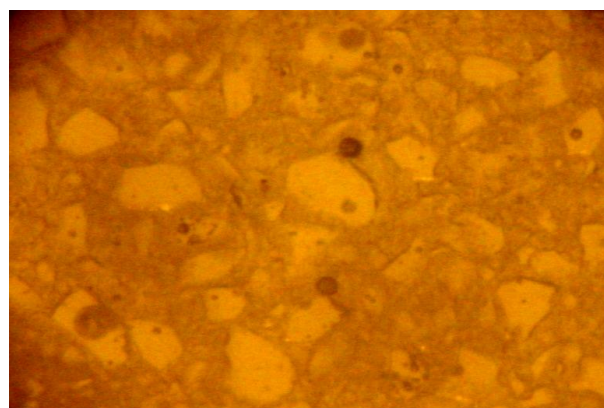


Figure 5. Photomicrograph of the same fused silica sample after annealing showing fused silica grains free from carbon contaminants

3) Key properties of the fused silica product

a) Chemical Quality Factors

The fused product was white in its appearance, did not show any sign of cristoballite and/or quartzite in its XRD pattern, Fig.6 and its complete chemical analysis is depicted in Table 9. It is clear that fused product is of high purity with SiO₂ around 99.96% and total impurities around 50 ppm. Compared with some international fused silica products, Table 10, it matches the Chinese Grade A only with SiO₂ content 99.9 % and is relatively better than other USA (C-E Minerals, Minco, Horbison Walker, USA) or UK (Tilcon Co. UK) products [14]. In terms of its total impurity content, the Egyptian fused silica comes before “Vitrosil” product of C.E Minerals, USA.

TABLE VIII. EFFECT OF CHANGING FEED SIZE, FUSION TEMPERATURE AND HOLDING TIME OF THE WHITE SAND SAMPLE ON THE PHYSICAL PROPERTIES OF ITS FUSION PRODUCTS

Size, mm	Temp., °C	Time, min.	Silica Sand		
			Density, g/cm ³	Porosity, %	C. T. E * X 10 ⁻⁶ K ⁻¹
-0.6+0.25	1800	15	2.0503	6.80	0.66
-0.6+0.25	1850	15	2.0807	7.50	-0.51
-0.6+0.25	1880	15	2.1027	7.22	0.57
-0.6+0.25	1880	25	2.2490	6.95	0.60
-0.25+0.177	1880	25	2.1980	5.85	0.62

* Coefficient of Thermal Expansion x 10⁻⁶ K⁻¹

TABLE IX. CHEMICAL ANALYSIS OF THE FUSED SILICA PRODUCED BY THE TAMMAN FURNACE

Constituent	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Na ₂ O	CaO	K ₂ O	P ₂ O ₅	TiO ₂	MgO	MnO
%	99.96	0.022	0.004	0.004	0.003	0.002	0.002	0.008	0.001	0.004

TABLE X. COMPARISON OF FUSED SILICA PRODUCTS, [14]

Properties	Tilcon UK	Linkason Enterprises China	Harbison-Walker USA
Brand name	Fused Silica	Fused silica	Vitreosil
Grade	-	Grade A 99.9% Grade B 99.8% Grade C 99.6%	-
Chemical	-	-	-
SiO ₂	99.5	99.6-99.9%	99.4
Al ₂ O ₃	0.25	0.2	0.40
Fe ₂ O ₃	0.1	0.1-0.5	0.10
CaO	-	0.02	0.10
MgO	-	0.02	0.10
TiO ₂	0.03	0.03	0.02
Na ₂ O	-	0.05	-
K ₂ O	-	0.05	0.03
Li ₂ O	-	-	-
Physical S.G. g/cm ³	2.2	<2.23	2.18

Under the optimum fusion conditions of 1850 °C and 45 min holding time after fusion process is completed for unbeneficiated silica, its XRD pattern show quartzite peaks at 2θ =26.61° indicating improper fusion, Fig. 6. At these high temperatures, any trace metal impurities such as Cr, Fe, Cu, Mn, Ni and Ti migrate to the surface of the raw materials, cover it and prevent it from complete fusion.

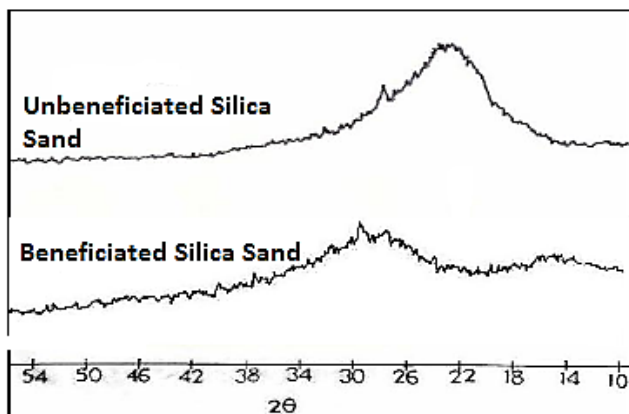


Figure 6. XRD pattern of fused silica produced from unbeneficiated and beneficiated raw material

The XRD pattern of the fused silica, produced from beneficiated raw materials, show almost complete fusion and absence of any quartz or cristoballite peaks in the fused silica

sample, (Fig. 6). However, the XRD pattern of the fused product of the beneficiated silica, as compared with the unbeneficiated silica powder sample, endorses the role of raw materials beneficiation to produce high quality end products.

IV. CONCLUSIONS

- The amenability of a white sand sample to beneficiation to improve its SiO₂ content was found to be inevitable prior the fusion process.

- Attrition scrubbing followed by a combined flotation-magnetic separation circuit, a final sand concentrate assaying 0.0059% Fe₂O₃ and 0.015% Al₂O₃ was obtained with an overall recovery of 70%.

-Chemical leaching of this concentrate, by using HCl and stannous chloride at 50-60°C, led to the production of ultra-pure sand concentrate having 0.0043% Fe₂O₃.

-Fused silica was prepared using either an arc furnace or an electrical resistance heating Tamman furnace.

-The arc furnace product was greyish brown in colour and coated with carbon dissipated from the graphite hearth and the electrodes affected its chemical constitution and physical properties. -Product of the electrical Tamman furnace was clear and exhibited excellent physico- mechanical properties that satisfy very well the requirements for high temperature refractory materials, special refractory industries and as filler for epoxy molded resins.

- However, fused silica, substantially, increases the value of the primary materials.

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