TEXHIKA ТА МЕТОДИ ЕКСПЕРИМЕНТУ ENGINEERING AND METHODS OF EXPERIMENT

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ASSESSMENT OF A POLYMERIC COMPOSITE AS A RADIATION ATTENUATOR AND A RESTORATION MORTAR FOR CRACKING IN BIOLOGICAL SHIELDS

This work is dedicated to figuring out robust epoxy/magnetite/boron carbide (EP/Mag/B₄C) composite for radiation attenuation at multiple applications related to nuclear installations, as well as restoration mortar for cracking developed in concrete biological shields. The mechanical properties (flexural, compressive, and impact strengths) and the physical properties (water absorption, porosity, and dry bulk density), each, have been performed to label the composite integrity for practical application. In practice, attenuation properties have been performed by using a collimated beam emitted from spontaneous fission ²⁵²Cf (100 μ g) neutron source and neutron gamma spectrometer with stilbene scintillator. The pulse shape discrimination technique which would come of the zero cross over method was used to measure the fast neutron and gamma-ray spectra. Thermal neutron fluxes have been measured by using the thermal neutron detection system and the BF-3 detector. The attenuation parameters: precisely, macroscopic effective removal cross-sections Σ_R (cm⁻¹), macroscopic cross-sections Σ (cm⁻¹), and total attenuation coefficients μ (cm⁻¹) of fast and thermal neutrons and total gamma-rays respectively were evaluated using the attenuation relations. Also, the MCNP5 code and MERCSF-N program have been used to compute the parameters theoretically. When applicable, measured and calculated results were compared, and it tells us a comprehensive agreement.

Keywords: biological shield, composite, neutron and gamma-ray spectra, attenuation parameters, MCNP code.

1. Introduction

Any concrete structure may develop structural or non-structural cracks either by aging or by any other cause. Cracks in concrete foundations greater than approximately 1 - 2 mm require sealing/injection to prevent the ingress of moisture, oxygen, and other materials [1, 2]. Also, the nuclear facilities' biological shield concrete structures can deteriorate in one way or another by the impact of increasing temperature as well as radiation exposure. It is quite right to say that irradiation of concrete to nuclear radiation leads to temperature increase, which affects the mechanical, physical, and radiation shielding properties accordingly. Cracks, chips, and breaks or flaking in concrete are not only unsightly but rather, they can lead to further deterioration to the surface [3, 4].

To repair concrete, it is important to select the right repair product, because not all products are suited for all purposes. An ideal product suited for general patching may not work well on small cracks and fractures.

Prevailing of polymer consumption encourages the growth of dispersed fillers incorporation to minimize the cost, besides the increase in strength and stiffness of the end composite. The composite's characteristic stems from matrix-filler interaction. Each constituent participates in rendering the composite material performance [5, 6].

Epoxy resin provides permanent bonding even for the smallest cracks and cracks ramifications in concrete and masonry – thanks to its high adhesiveness as well as its overall strength. The main advantage of epoxies is their compressive strength, which exceeds most of the concretes at 12,000 psi or greater. On the other hand, epoxies cure very slowly, generally taking hours to harden. This can be an advantage because it allows time for the epoxy to flow into even the smallest crevices [7].

Selecting a radiation shielding media is a rather complex matter since it is supposed to deal with different types of radiation. Gamma-rays as well as neutrons of different energies. Therefore, we should attain a combination of heavy, light, and thermal neutron absorber components. Generally, heavy components (elements) deal with gamma-rays as well as fast neutrons through (inelastic scattering). In addition to that, light components (elements) would be responsible for intermediate neutrons through (elastic scattering). Finally, thermal neutron absorbers: they have a dual role as thermal neutron absorbers and indirectly preventing gammas that could have happened

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through (heavy elements "Fe") absorption of thermal neutrons [12]. In our work, epoxide formulations provide good neutron attenuation; it has been suggested that it might be also suitable as shields for gamma and X-rays by adding heavy minerals [8]. Magnetite (Fe₃O₄) having a high density (4.9 - 5.2 g/cm^3) is an effective shielding material for neutrons and gammarays [9, 10]. In addition to such high-density aggregate, additives containing boron are sometimes used to improve shielding properties, where boron has been induced as a neutron absorber in various materials in addition to concrete as a general practice; however, some boron compounds influence cement hydration and retard setting if added with high loading fractions to concrete castings, which does not exist in the case of the polymer composite. For instance, borated graphite (a mixture of elemental boron and graphite) has been used in fast reactor shields. Boron has also been added to steel for shield structures to reduce secondary gamma-ray production. In addition to that, boral - consisting of boron carbide and aluminum – has been used for shielding purposes [11, 12].

To restore concrete structures to their original strength, the formula is suggested as a repair mortar for developing cracks in the biological shields. This mortar will be satisfying the requirements of improved plastics, such as high mechanical strength, adhesiveness, and reasonable physical properties, besides its main role in radiation shielding for this application.

Recently, many researchers have studied the effect of the dispersed fillers in a variety of polymers, which satisfies the requirements for radiation shielding [13, 14, 17]. The prepared composite (EP/Mag/B₄C) can be utilized in radiation attenuation with different applications, as an example, for shipping and storage of radionuclide materials for many mobile and stationary sources. Also, it can be used as portable radiation shielding, for example, medical lining for the walls of diagnostic and radiotherapy rooms. Besides, it might foresee applications for detector shields, neutron guides, valves, and pipes [15]. As well, this composite could be applied as rendering/plastering mortar or even coating for shielding structures present in nuclear foundations as a possible example for the many epoxy composites practical uses [16].

The present study has the primary aim of using

epoxy, magnetite, and boron carbide to prepare $EP/Mag/B_4C$ formulation for the construction of radiation attenuation shield used for a variety of applications, as well as a mortar for developing cracks in biological concrete shields. Measurements have been carried out to come through their mechanical, physical, and radiation attenuation properties. Also, theoretical calculations have been achieved using MCNP5 and MERCSF-N programs.

2. Experimental procedure

2.1. Materials and sample preparation

The concerned composite epoxy/magnetite/boron carbide (EP/Mag/B₄C) was prepared with definite weights in terms of constituents to provide suitable mechanical, physical, and attenuation properties. The standard Bisphenol-A based Epoxy resin (EP) of the commercial name (DGEBA DER 331 product of DOW Chemical Company-USA, with technical purity 95% and epoxide weight 182-192) hardened by polyoxyporopylendiamine (Cetepox 1465 H product of Chemical & Technologies for Polymers Co., Egypt) was used to work out the composite base. Magnetite supplied by the Nuclear Materials Authority (El Kattameya, Egypt) in the form of a fine black powder of mesh size 500 µm obtained by physical separation from the coastal black sand which was accumulated by the precipitation of weathered heavy minerals along the Nile Valley pathway and boron carbide (B_4C) chemical of -60 + 230 mesh (a product of Sigma-Aldrich Ltd, England) were used as composite fillers. First, appropriate Teflon molds were glued to the lab planchettes. Cylindrical molds (10 cm in diameter and \approx 5 cm in thickness) acting for radiation attenuation measurements and molds of ASTM dimensions for the required mechanical and physical tests. The formulation ingredients (resin – curing agent - fillers) were weighed with an electronic balance and were then mingled together to obtain a homogenous mixture. The constituents where: EP = 15 %, Mag = 75 % and $B_4C = 10 \%$, which stands for the EP/Mag/B₄C composite. The elemental composition of both blank and filled formulations was evaluated by the aid of chemical pamphlets and magnetite supplier analysis is given in Table 1.

Sample	Н	В	С	Ν	0	Na	Mg	Si	K	Ca	Fe
EP	00.066		0.6745	0.0285	0.2310						
EP/Mag/B ₄ C	0.012170	0.077010	0.128500	0.004050	0.276409	0.007300	0.002300	0.057990	0.000561	0.047310	0.386400

Table 1. Chemical composition by weight fraction of the investigated formulations

We note here that, the sample workability is governed by limitations imposed by the ability of a liquid component (resin + hardener) to accept the reasonable dry component (fillers) percentage without violating the mortar's consistency and fluidity. Afterward, the mixture was degassed to allow the entrapped air bubbles to be released. Finally, it was poured with great care in the specimen's Teflon molds. After curing, the samples were released from molds and left for more than one week to allow the cross-linking process to propagate. The expected cross-linkages were completed. They were furthermore shaped with steel adaptation frames to the final dimensions.

2.2. Mechanical and physical tests

The mechanical tests: compressive, bending and Charpy impact strengths have been achieved at room temperature at the National Institute for Standards (NIS) of Egypt. The 400 KN ZWICK Universal Testing Machine (No 15376), with calibrated cell and Charpy impact hammer, were used to perform such experimental tests according to the ASTM D-695, ASTM D-790, and ASTM D-256 designations [18 -20]. Dry bulk density, water absorption, and total porosity were measured depending on the ASTM designation (D 570-81) and ASTM designation (C 948-81) re-approved 2001 [21, 22].

2.3. Fast neutron and gamma-ray measurements

Fast neutron and total gamma-ray (primary + secondary) spectra have been measured behind cylindrical epoxy blank (EP) and epoxy/magnetite/boron carbide (EP/Mag/B₄C) composite samples of 10 cmdiameter and different thicknesses. Measured spectra have been carried out by means of a collimated beam emitted from the spontaneous fission 252 Cf (100 μ g – June 2001) neutron source and neutron gamma spectrometer with cylindrical stilbene scintillator $(\emptyset = 4 \text{ cm and thickness} = 4 \text{ cm})$. The collimated beam was provided by the narrow beam experimental facility that consisted of the radioactive source + collimator - samples holder - detector + collimator. The arrangement layout is shown elsewhere [13]. The purpose of the beam – detector collimation is to provide a beam of a specific intensity and to divert side scattered radiations from the detector path which would enhance the discrimination capability. The spectrometer components, set up, and discrimination technique happened to be explained in detail elsewhere [23 - 25]. Spectrometer discrimination, linearity, and energy scaling were checked before taking measurements by accumulating spectra of ²²Na, ¹³⁷Cs, ⁶⁰Co, and Pu-a-Be sources. Measured pulse amplitude distributions of the recoil protons or electrons were converted into energy spectra of fast neutrons or gammarays using two unfolding codes NSPEC and GSPEC originated on the double differentiation and matrix correction methods for neutrons and gamma-rays respectively [26, 27].

2.4. Slow neutron measurements

Slow neutron fluxes have been measured behind the concerned epoxy blank (EP) and (EP/Mag/B₄C) formulations with different thicknesses using the thermal neutron detection system plugged with the BF-3 (LND-20354) detector. The same experimental layout in section 2.3 was used where BF-3 tube was introduced into the detector collimator given an operating voltage of 1600 V. Output pulses drawn from the BF-3 were fed to the preamplifier, then to the amplifier type ORTEC 572A. The magnified pulses were fed into the PC incorporating a TRUMP 8K/2K data acquisition card. The diagram of the measuring system components was shown elsewhere [28]. The slow neutron (0 - 1000 eV) fluxes were measured by integrating the specified net area under the peaks 2.31 and 2.79 MeV [29].

3. Theoretical calculations

3.1. MCNP calculation

MCNP5 computer code was used to reflect a three-dimensional model that matches the experimental setup. For the source term, a built-in card associated with ²⁵²Cf spontaneous fission neutron source is quite right to perform the source spectrum. At the detection term, both detectors' interactive regions, stilbene, and BF-3 were introduced to the code and the DXTRAN sphere was embedded within such detection mediums to improve the results' accuracy.

The code is to run using analytical methods starting from the source term till the outer shell of such sphere and continues using the MCNP technique to the central shell. To perform fast and thermal neutrons and gamma-ray calculations, the code was first to run in NP mode followed by P mode. Neutron, as well as induced gamma-ray tallies, was obtained from the first runs while pure photon results could be deduced from the second runs. The total photon term results from the summation of both pure and induced terms. A number of 10^8 NP and 10^9 P histories were accumulated to perform the calculations, in which tallies were scaled to ²⁵²Cf strength using appropriate tally multiplication cards [30]. The MCNP computer code with (95 energy groups) for fast neutrons and (55 energy groups) for gamma-rays was used to calculate, theoretically, the spectra behind the two formulations (EP and EP/Mag/B₄C) of thicknesses 19.02 and 20.56 cm for neutrons and gamma-rays, respectively. The obtained spectra are presented in Figs. 7 - 10.

3.2. Macroscopic effective removal cross-section of fast neutrons

The macroscopic effective removal cross-sections of fast neutrons $\Sigma_{\rm R}$ (cm⁻¹) through both EP and EP/Mag/B₄C formulations have been calculated using the MERCSF-N computer program. In principle, the program had been constructed, verified, and applied for calculating the macroscopic effective removal cross-section $\Sigma_{\rm R}$ (cm⁻¹) of fast neutrons through homogeneous mixtures, compounds, concretes, and composites. To achieve the calculations, atomic masses, and mass removal cross-sections Σ_R/ρ (cm^2/g) for a collection of elements were fed to the program as input physical data. MERCSF-N program; description and confirmation, in details with regards are given elsewhere [31]. The program output illustrates the macroscopic effective removal crosssection $\Sigma_{\rm R}$ (cm⁻¹) of fast neutrons through the concerned target. The applications of the program are given in numerous publications, for instance [13, 14]. Results for EP and EP/Mag/B₄C formulations are presented in Table 4.

4. Results and discussion

4.1. Mechanical properties

The epoxy blank (EP) and epoxy/magnetite/boron carbide (EP/Mag/B₄C) mechanical strengths are shown in Table 2. It shows the values of the mechanical properties decrease for the EP/Mag/B₄C than EP and this is due to the fillers loading fraction. The filler load leads to particle agglomeration which affects the composite consistency and, consequently, lower filler-resin matrix bonding. In other words, particle loading transcends the critical level, called mechanical percolation.

Table 2. Measured mechanical properties for EP and EP/Mag/B₄C formulations

	Bendi	ng	Compressi	Impact		
Sample	Sample Flexural strength, MPa		Compressive strength, MPa	Modulus of elasticity, MPa	Impact strength, J/m	
EP	85	_	90	_	-	
EP/Mag/B ₄ C	31.03	9363.96	62.82	2454	142.97	

4.2. Physical properties

Water absorption, porosity, and dry bulk density for the concerned epoxy blank (EP) and epoxy/magnetite/boron carbide (EP/Mag/B₄C) composite are presented in Table 3. It is noticed that the EP/Mag/B₄C composite has higher values than those for epoxy blank (EP). It is to say that this may be attributed to the fact that filler incorporation affects composite cross-linking since it introduces pores to the matrix. By comparison to published data [13], the obtained results are reasonable in terms of practical applications.

Table 3. Measured	physical properties	for EP and EP/Mag/B4	C formulations
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Sample	Water absorption, %	Porosity, %	Dry bulk density, g·cm ⁻³
EP	0.0106	0.0124	1.16
EP/Mag/B ₄ C	0.0389	0.0660	2.995

4.3. Attenuation results

The measured pulse amplitude distributions of recoil protons and electrons were converted to energy distributions (spectra) of neutrons and gamma-rays, respectively. The observed spectra of fast neutrons exiting epoxy blank (EP) ($\rho = 1.116 \text{ g/cm}^3$) with thicknesses; bare (0), 4.36, 8.50 ... and 19.02 cm, and epoxy/magnetite/boron carbide $(EP/Mag/B_4C)$ $(\rho = 2.995 \text{ g/cm}^3)$ with thicknesses; bare (0), 4.30, 8.56 ... and 20.56 cm, respectively, are displayed in Figs. 1 and 2. The general trend for the intensity of the neutron spectra is for a decrease versus the increase in energy and formula thickness. Displayed spectra almost have the same shape and profile for both formulations. It is clear, the spectra do not show the buildup of neutrons at low energies, and this may be attributed to the fact that removal of fast neutrons by inelastic scattering is not so effective. Neutrons of energy above the threshold of inelastic scattering are low at the incident beam. It is observed that the spectra depend on the sample thicknesses for neutrons of energies within 1 to 7 MeV. For energies above 7 to 9 MeV, spectra do not follow regular decrease as detection uncertainty varies for such high energies as shown in both figures.

In Fig. 2, the magnetite effect on the neutron attenuation is clear for all thicknesses and at all energies since the mineral, with mainly iron component, reduces flux through successive cascades of inelastic reactions along the sample. Also, we might need to point out; spectra nearly close-up with increasing the thicknesses for all energies in both figures. This may be attributed to the reduction of the neutron yield with the extension of the formula thickness.



The total gamma-ray (primary + secondary) spectra behind the concerned formulations with the same thicknesses are displayed in Figs. 3 and 4. The displayed spectra show similarity in shape and profile for all measured thicknesses. The spectra have closely similar profiles: A decrease in intensity with an increase in the photon energy and composite thicknesses. The maxima of the gamma-ray energy working at about 2.225 MeV (in both curves) refers to the contribution of the captured gamma-rays due to the absorption of slow neutrons by hydrogen atoms. It is clear that EP/Mag/B₄C have spectra lower than those for epoxy blank (EP), and this may be attributed to the magnetite and boron carbide effect, i.e. the composite density.

Furthermore, measured fluxes have been integrated over the observed energy range (0.8 to 9 MeV) and (0.4 to 6 MeV) for fast neutrons and gamma-rays, respectively. For slow neutrons, fluxes have been integrated under the peaks located at 2.31 and 2.79 MeV. The integrated values are plotted against the thicknesses and are graphed in Figs. 5 and 6. The attenuation curves Fig. 5 show that fast neutrons and gamma-rays fluxes intensity decrease exponentially with increasing composite thicknesses. The fall-off is the least for gamma rays and greatest for fast neutrons and both formulations. For EP/Mag/B₄C this may be attributed to the fact that the loss of gamma-rays is compensated by fast and slow neutrons interaction, namely, to the addition to photons economy that comes through neutron inelastic scattering by iron present in magnetite, in addition to the rendered thermal neutron reaction with iron (which gives hard gammas). And soft gammas from thermal neutron reaction with boron present in B_4C . Also, for EP, the fast neutron will react with epoxy components (mainly carbon and oxygen) through elastic scattering, giving gammas indirectly. In addition to the 2.23 MeV radiative capture gamma-rays from thermal neutron reaction with hydrogen; rendering, finally, to increase in gamma-rays in total. All this results in lower attenuation for gamma-rays. The usual attenuation relations were used to obtain the macroscopic effective removal cross-sections $\Sigma_{\rm R}$ (cm⁻¹) and total attenuation coefficients μ (cm⁻¹). Fig. 6 presents the attenuation relations of slow neutrons where the fluxes intensity decreases exponentially with increasing composite thicknesses. Such relations were used to evaluate the macroscopic cross-sections Σ (cm⁻¹). The absorption effect is clear for slow neutrons by B_4C in the case of EP/Mag/B₄C composite.



behind EP and EP/Mag/B₄C formulations.

The obtained experimental and theoretical (MCNP5 and MERCSF-N) shielding attenuation parameters, macroscopic effective removal cross-sections $\Sigma_{\rm R}$ (cm⁻¹), macroscopic cross-sections Σ (cm⁻¹), and total attenuation coefficients μ (cm⁻¹) of fast and

slow neutrons and total gamma-rays respectively are shown in Table 4. In addition, it is noticed that measured and theoretically evaluated parameters are close and this confirms the obtained results, as well as the measurement and calculation methods.

Table 4. Measured and calculated attenuation parameters for EP and EP/Mag/B₄C formulations

Attenuation parameter, cm ⁻¹	EP	EP/Mag/B ₄ C
$\Sigma_{ m R-Meas}$	0.09531 ± 0.00373	0.11295 ± 0.00196
$\Sigma_{ m R-MCNP}$	0.12348 ± 0.00271	0.13853 ± 0.00396
$\Sigma_{ m R-MERCSF}$	0.10335	0.12060
μ_{Meas}	0.06353 ± 0.00108	0.09457 ± 0.00185
$\mu_{ m MCNP}$	0.05094 ± 0.00456	0.08245 ± 0.00255
$\Sigma_{ m Meas}$	0.05373 ± 0.01557	0.05832 ± 0.00722
Σ _{MCNP}	0.05551 ± 0.00146	0.05736 ± 0.00299





Figs. 7 - 10 present a comparison between the experimentally measured spectra and theoretically calculated ones using MCNP5 code for fast neutrons (95 energy groups) and total gamma-rays (55 energy



Fig. 8. Measured and calculated (MCNP - 95 energy groups) fast neutron spectra behind EP/Mag/B₄C sample.

groups) behind the two formulations (EP and EP/Mag/B₄C) of thicknesses 19.02 and 20.56 cm, respectively. It shows that there is a fair resemblance between experimental and theoretical spectra.



groups) total gamma spectra behind EP sample.

5. Conclusions

From the measured and calculated results for EP and EP/Mag/B₄C formulations, it can be concluded that the measured mechanical properties for EP/Mag/B₄C composite are less than EP formula, while the physical properties for EP/Mag/B₄C are higher versus EP. However, the obtained measurement results are reasonable in terms of practical applications. Besides, fast neutron and total gamma-ray spectra decrease with the increase of energy and formulation thicknesses. This supports the general rules of shielding, which states that a combination of materials is required; heavy material, light material, and thermal neutron absorbing agent would be necessary. The attenuation relations for fast and slow neutrons and total gamma-rays show the fluxes to exponentially decrease with the increase of the formulation thicknesses. The measured and calculated attenuation parameters; $\Sigma_{\rm R}$ (cm⁻¹), Σ (cm⁻¹), and μ (cm⁻¹) for fast



Fig. 10. Measured and calculated (MCNP - 55 energy groups) total gamma spectra behind EP/Mag/B₄C sample.

and thermal neutrons and total gamma-rays respectively through EP and EP/Mag/B₄C are in reasonable agreement, which confirms the measurement and calculation methods. As well, experimentally, and theoretically MCNP5 obtained spectra of fast neutrons and total gamma-rays behind certain thicknesses of formulations (EP and EP/Mag/B₄C) display a fair resemblance, which confirms the experimental and theoretical methods. The obtained attenuation parameters in Table 4, either measured or calculated, are all giving preference to the EP/Mag/B₄C composite for attenuation of fast, thermal neutrons and gamma-rays. This supports, the created EP/Mag/B₄C composite offers good mechanical, physical, and attenuation properties for many shielding applications, and could be proposed as an injecting mortar for cracks in biological shields. However, further investigations are planned for different filler ratios, for comparison purposes, in order to reach optimal shielding properties.

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ОЦІНКА ПОЛІМЕРНОГО КОМПОЗИТА ЯК ОСЛАБЛЮВАЧА РАДІАЦІЇ ТА РЕСТАВРАЦІЙНОГО РОЗЧИНУ ДЛЯ ВІДНОВЛЕННЯ РОЗТРІСКУВАНЬ У БІОЛОГІЧНОМУ ЗАХИСТІ

Ця робота присвячена дослідженню стійкого композиційного матеріалу на основі епоксиду/магнетиту/карбіду бору (ЕР/Маg/B₄C) для ослаблення радіаційного випромінювання при багатьох застосуваннях, пов'язаних з ядерними установками, а також розчину для усунення розтріскувань у бетонному біологічному захисті. Було досліджено механічні властивості (міцність на вигин, стиск та ударна міцність) та фізичні властивості (водопоглинання, пористість та насипна щільність) для визначення придатності композиту для практичного застосування. Ослаблення радіації вимірювалося за допомогою колімованих пучків нейтронів, що випромінювалися при спонтанному поділі ²⁵²Cf (100 мкг), та спектрометра гамма-квантів і нейтронів із стильбеновим сцинтилятором. Використовувалася дискримінація за формою імпульсу з використанням методу нульового перетину для отримання спектрів швидких нейтронів та гамма-квантів. Потоки теплових нейтронів вимірювали за допомогою системи детектування теплових нейтронів з детектором BF-3. Параметри ослаблення, а саме: макроскопічні ефективні поперечні перерізи Σ_R (см⁻¹), макроскопічні перерізи Σ (см⁻¹) та загальні коефіцієнти ослаблення μ (см⁻¹) швидких та теплових нейтронів та гамма-квантів оцінювалися за допомогою відповідних співвідношень. Для теоретичного обчислення параметрів використовували програми MCNP5 та MERCSF-N. Виміряні та розраховані результати порівнювалися, коли це було можливо, і порівняння свідчить про їхню гарну згоду.

Ключові слова: біологічний захист, композит, спектри нейтронів та гамма-квантів, параметри ослаблення, програма MCNP.

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