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Review / Przegląd

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Aspects of nanomaterials for civil and military applications. Part 1. Origins, characteristics and fabrication methods *Aspekty nanomateriałów w zastosowaniach cywilnych i militarnych. Część I. Pochodzenie, charakterystyka i metody otrzymywania*

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Abstract: *The study presents general aspects of highly dispersed nano scale materials including their origins, structure, classification, properties and production methods. In the last two decades, the unique properties and phenomena observed for those material has revolutionized the industry with a significant increase in research and the scope of practical applications of nanotechnology in every aspect of our lives.*

Streszczenie: *W pracy przedstawiono podstawowe aspekty materiałowo wysokim stopniu zdyspersgowania w skali nanometrycznej obejmujące pochodzenie, budowę i klasyfikację, wykazywane właściwości oraz ich metody wytwarzania. Osobliwe właściwości i zjawiska wykazywane przez te materiały sprawiły, że w ostatnich dwóch dekadach jesteśmy świadkami rewolucji materiałowej. Świadczy o tym zarówno i istotny wzrost intensywności prowadzonych prac badawczych jak i rosnący zakres możliwości praktycznego stosowania osiągnięć nanotechnologii we wszystkich dziedzinach naszego życia.*

Keywords: *high-energy nanomaterials, origins, properties, synthesis methods*

Słowa kluczowe: *nanomateriały wysokoenergetyczne, pochodzenie, właściwości, synteza*

1. Introduction

A concept of miniaturization of physical objects, presented in 1959 by eminent physicist R.N. Feynman, gave rise to a new branch of science referred to as nanoscience (*nano* – from the Greek word “*nanos*” meaning “dwarf”) [1]. It is an interdisciplinary science combining physics, chemistry, biology, IT, mechanics and many other areas of science. It studies the phenomena and properties arising from the structure of matter on an atomic, molecular and macro-molecular level, particularly in the 1-100 nm region, which differ significantly from those

observed on a micro or macro scale [2-5]. The symbol ‘nm’ (nanometre) means an aliquot of 1 m, a basic length unit in the SI system, corresponding to one billionth of a metre, whereas μ (micro) is an aliquot corresponding to one millionth of a metre:

- $1 \text{ nm} = 10^{-9} \text{ m}$ (0.000000001 m),
- $1 \text{ }\mu\text{m} = 10^{-6} \text{ m}$ (0.000001 m).

Usually, nanoparticles (NPs) classified as nanomaterials are 100,000 to 1,000,000 times smaller than 1 mm. The concept of ‘nanoparticle’ first appeared in scientific literature in 1974 [3]. For comparison, these structures are 200 times smaller than human. On a scale of an elephant to an electron, nanomaterials range from bacteria to single atoms. Figure 1 illustrates the dimensions. A human hair visible with the naked eye, has a diameter of 80,000 nm.

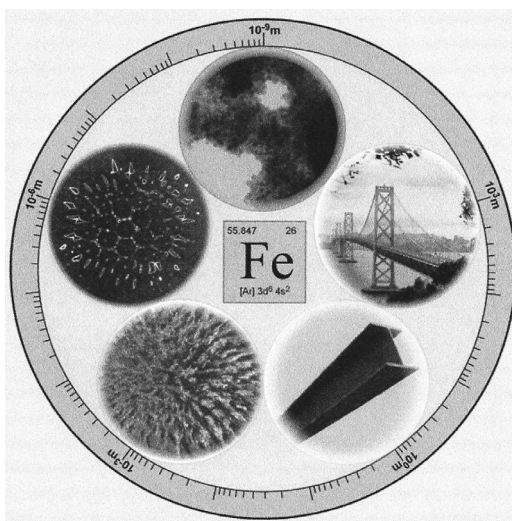


Figure 1. Dimension range from a bridge to a single atom [6]

The developments in nanoscience are used in nanotechnology, also referred to as engineering on a nanoscale or innovative engineering. It is a branch of science dealing with intentional and controlled modelling and fabricating of objects in the nanoscale in the form of materials, composites, systems or devices showing unique properties and practical applicability [7-11].

The products of nanotechnology are referred to as nanomaterials (nanoobjects). In accordance with the recommendations published in the Official Journal of the European Union in October 2001, the term is defined as a naturally occurring, accidentally or intentionally fabricated material containing free particles and/or aggregated particles (clusters) in which at least 50% particles in the numerical particle size distribution has one or more dimensions (length, width, height) between 1 and 100 nm [12]. This range may also apply to the size of a domain as a basic unit of the microstructure. It was described as such in the recommended EU definition, however, it is not physically justified. The case is similar for colloidal solutions, where the size of particles forming the colloidal system is between 1-1000 nm [13]. It means that in the subject literature, one definition covers nanomaterial made of particles for which at least one of three spatial dimensions is less than 100 nm, while another definition covers materials in which, at a specific material particle size, there are significant differences in physical and chemical properties compared to the properties of the same material at the micro and macro scale [8, 14].

The unique properties showed by nanomaterials has resulted in the dynamic development of nanoscience and nanotechnology over the last two decades. This is reflected not only in a rapidly growing number of published theoretical, experimental and revision studies and patents, but also a growing number of products containing

nanomaterials for practical use in almost every aspect of our lives.

The developments in nanotechnology, in particular the unique properties of its products (nanomaterials, nanoobjects, nanorobots) has made this technology a major focus of interest in the defence industry. Current solutions can be applied to virtually every aspect of the defence industry, from seemingly civil applications including ultralight footwear and clothing, through to improvements in the efficiency of high-energy materials, solid and liquid rocket propellants, means of communication and optical devices to nanorobots [11, 15-27].

Nanoscience, nanotechnology, nanotechnique, nanoparticles, nanomaterials, nanoobjects, nanomachines, nanothermites, nanoenergetics, nanoeconomy, nanobusiness are some of the terms with a **nano-** prefix being encountered more frequently in literature on materials, financial markets and media. International science conferences on the propagation of developments in nanoscience and nanotechnology are organized annually. The Annual World Nano-Economy Congress (WNEC) gathers leaders in nanotechnology and nanobusiness, showing the growing significance of this new industry. Quarterly bulletins have been published online since 1997 by the Advances Materials & Processes Technology Information Analysis Center (AMPTIAC) and include the latest information on advanced materials and innovative developments in nanoengineering [28]. Cyclical conferences on various aspects of nanoscience and nanotechnology are also organized in Poland [29].

2. Origins of nanomaterials

Many different structures found in animate and inanimate matter can be classified as nanomaterials [30-34]. Using their origin as a criterion, nanomaterials can be classified as:

- natural, with a large variety in the environment such a level as to form conditions depending on the weather, in the form of:
 - a) atmospheric aerosols, a product of complex physical and chemical processes in the earth's atmosphere and various combinations; the products are mostly aerosols of sulfates and nitrates the amount of which is dependent on geographical location [7, 35],
 - b) nanocrystals occurring in mineraloids formed as a result of processes occurring in the earth's crust, for example semi-precious opal [36],
 - c) different structures formed by organic nanomaterials including hide, horns, beaks, bones, feathers and claws [37],
 - d) natural colloids including milk, blood, paper or cotton fibres [35-37],
 - e) sea salt microcrystals formed by evaporation of sea spray, classified as atmospheric aerosols [35],
 - f) active volcanoes releasing volcanic dust [35],
 - g) products of biological processes of different bacteria genus, including bacterial cellulose (BC) [37],
 - h) forest fires [35],
 - i) sand storms generating dusts transported over large distances by air mass flow [35],
 - j) clays, including structures made of nanoplates [5];
- anthropogenic, including:
 - a) secondary, a product of accidental application, for example formed as a result of combustion or friction of organic material, *e.g.* amorphous carbon (C, soot) – a source of atmospheric nanoaerosols [35],
 - b) accidental, due to difficult to anticipate environmental conditions [38, 39],
 - c) intentionally generated for a specific purpose or product,
 - d) more and more advanced techniques; these feature a specific structure and show properties used in many industrial sectors [40-46].

Highly dispersed materials, currently classified as nanomaterials have been known since ancient times:

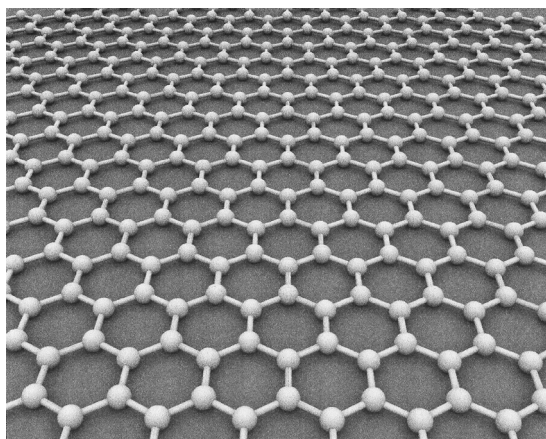
- different dyes, *i.e.* colorants:
 - a) inorganic (mineral) used in paints (*e.g.* ZnO, HgS, TiO₂, Fe₂O₃) and for dyeing glass and ceramics and colloidal enamels containing gold and/or silver; a method of preparing red gold sol also known as gold-tin purple was described in the 17th century by Glauber [30, 31, 35-38, 41],
 - b) organic (*e.g.* chlorophyll, indigo) used for dyeing cloth [3, 8, 18];

- cement obtained by the calcination of minerals (cement rock, limestone, clay) and grinding the agglomerate to obtain particle sizes between 3-100 μm ($1 \mu\text{m} = 10^{-6} \text{m}$) [3],
- amorphous carbon (C), soot obtained as a result of partial combustion of organic matter [47].

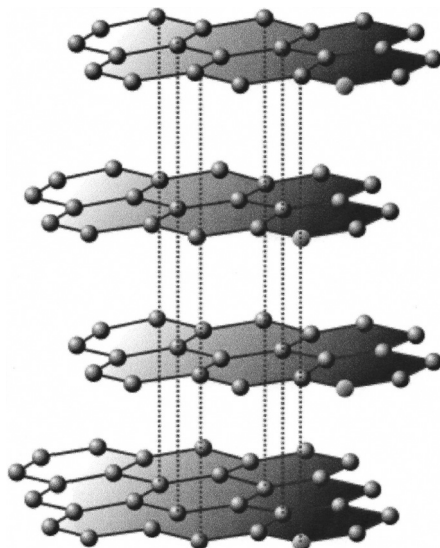
Examples of anthropogenic nanomaterials with a high potential for practical applications include:

- graphene (Fig. 2), characterized by a flat structure of carbon atoms (C) joined in hexagonal rings similar to a honeycomb, with the thickness of a single atom, classified as 2D structures; unique properties of this material, electrical, mechanical and other make it a major focus of interest in many industries [7, 9, 11, 40, 41, 48-50],
- fullerenes are materials with particles with an even number of carbon atoms (C) from 28 to approximately 1,500, forming closed, empty solids with chemical properties similar to aromatic hydrocarbons.

The most stable material in the fullerene group is C_{60} , also known as buckminsterfulleren or buckyballs [7, 21, 30, 31, 33, 50-55] (Fig. 3).



(a)



(b)

Figure 2. Structure: (a) graphene, (b) graphite [48]

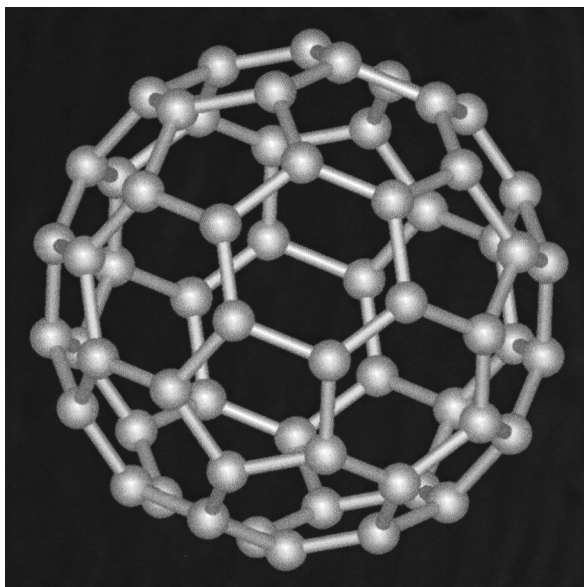


Figure 3. C₆₀ fullerene structure [55]

The fullerene molecules may be linked to polymer chains to obtain different composites used as lubricants or materials showing unique electrooptic properties [7, 23]. Introducing a different functional group yields catalytic systems with a highly developed surface area [3, 5, 10, 34, 42]. Almost any element from the periodic table and small particles of other chemical compounds can be encapsulated in fullerenes [7, 9, 23, 33, 42]. Similar properties can be found in clathrate compounds – hydrates of methane (CH₄), ethane (C₂H₆), ethylene (C₂H₄) and nitrogen(I) oxide (N₂O) [35, 39].

Available fullerene modifications can be used to form the following:

- exohedral fullerenes – (surface-modified) with different functional groups linked to the surface, *e.g.* addition of hydroxyl groups (–OH) yields fullerenols [32-34, 42],
- endohedral fullerenes – (internal-modified) with atoms or particles encapsulated within their structure [32-34, 41],
- heterofullerenes – at least one atom of carbon (C) is replaced by another element, *e.g.* nitrogen (N):
 - a) carbon nanotubes (CNTs) are a specific type of fullerene isomers (Fig. 4) showing good superconducting and semiconducting properties [51-55],
 - b) germanene, an allotrope of germanium (Ge) featuring a flat 2D structure similar to graphene and silicene showing unique semiconducting properties (field-effect transistors) and optical properties, making it a subject of interest to the electronics industry (electronic components) [56],
 - c) silicene, an allotrope of silicon (Si) featuring a flat 2D structure similar to grapheme [49, 56, 57],
 - d) tinene, an allotrope of tin (Sn) featuring a flat 2D structure showing superconducting properties [49, 58, 59].

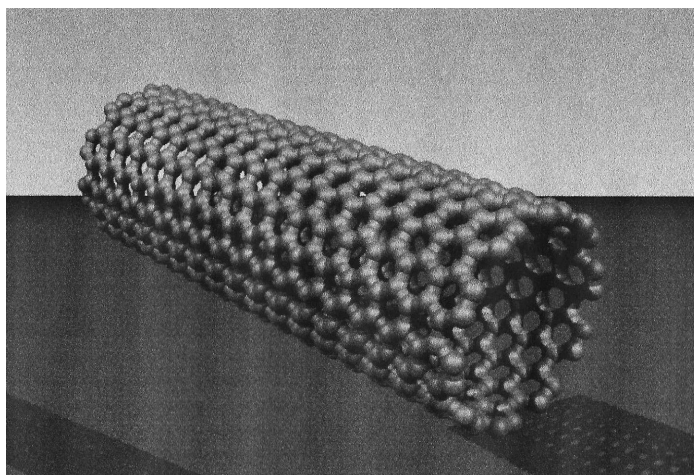


Figure 4. Carbon nanotube structure [52]

3. Structure and classification of nanomaterials

The nanomaterial structure is treated as a specific arrangement of different objects, including kernels, particles, crystallites, clusters *etc.* The complexity of the system means that, to determine the quantitative characteristics of the structure, the characteristic features of the objects must be determined, including:

- the number of objects,
- the dimensions (sizes) of objects, with different threshold values, usually related to new properties or specific phenomena,
- the share of specific objects in a volumetric distribution,
- the shapes of objects,
- the arrangement of objects [3, 4, 8, 13, 23, 30-34, 41, 47, 59].

Key criteria in the classification of nanomaterials including pure metals, metal oxides and alloys, ceramic materials, glass or elemental carbon clusters (C) are the dimensions, shape and chemical composition of their crystallites. The nanomaterials can be classified by size as follows [8, 30, 42, 43, 53, 60]:

- zero dimensional (0D), including quantum dots, clusters, nanocrystals, *e.g.* silver (Ag) nanoparticles [60-62],
- one dimensional (1D) with nano-size in two perpendicular directions, *e.g.* metal nano wires, nano rods [63], semiconductors [64, 65], polymer nanofibers [42, 52], plate fillers used in different industry sectors [7],
- two dimensional (2D) including nano-layers (sheets), single-phase, multi-phase or composites containing nanoparticles, *e.g.* carbon nanotubes (CNTs) [52-54],
- three-dimensional (3D), homo- and heterogenous monocrystalline nanomaterials containing crystalline domains or phase clusters or composites containing nanoparticles [7, 8, 30-33].

If a specific nanostructure features limited conductivity, it is referred to as:

- a quantum well if one of the dimensions is in the nanoscale (*i.e.* 1-100 nm) [7, 33],
- quantum wire if two dimensions are in the nanoscale [7, 63],
- quantum dots if all three dimensions are in the nanoscale [8, 63].

The nanomaterials can be classified by crystallite shape as follows:

- rod-shaped with rods in the nanoscale, classified as one-dimensional (1D) systems,
- layer-shaped with flat crystallites with nanotube thickness, classified as two-dimensional (2D) systems,
- equiaxed crystallites, sphere-shaped in the nanoscale classified as three-dimensional (3D) systems [3, 8, 13, 30-34, 40, 41].

Nanomaterials can be classified by chemical composition, as follows:

- containing crystals and/or crystallites with identical chemical composition,

- containing crystallites with different chemical composition,
- containing crystallites and/or phases in crystallite grains with different chemical composition,
- containing nanocrystallites with different chemical composition in their matrices [3, 13, 30-41].

Coating the material with a protective layer allows a new class of composite material with extensive practical applications to be obtained, featuring the following nanostructures:

- core-shell,
- yolk-shell [8, 32, 41].

The coatings can be modified by coupling with other chemical species to introduce functional groups to the nanostructure, giving it specific properties with a high potential for practical applications [41]. Core-shell nanoparticles include at least two different phases with different chemical composition and/or structure. Those properties mean that the material is a hybrid, showing both core and shell properties [32, 41].

4. Properties

Miniaturization of the dimensions of particles occurring in a conventional microstructure (macro- and microparticles) to the nanoscale, yields products showing unique properties and phenomena which are significantly different than those observed for the original material [8, 13, 30, 32, 41, 44].

The observed changes in physical and chemical properties are usually caused by two factors, *i.e.*:

- quantum constraints of electrons in small matter particles,
- increased ratio of surface and subsurface atoms or ions to atoms inside the particle [8, 14, 43].

This is indicated by the large specific surface area of nanomaterials and the increase in the number of unsaturated coordination positions, defects and stresses in the crystal lattice [8, 30]. A highly developed surface area results in an increase in chemical reactivity of the particles, usually reacting with their surroundings. The high degree of dispersion of nanoparticles means that they can be carried over large distances and are treated *a priori* as a serious hazard to the environment [66-68]. The small size of nanoparticles means that they may not only easily enter any living organism with inhalation but also cross biological borders [67].

Among the currently known properties of nanomaterials, the following unique properties and phenomena are noteworthy:

- chemical, including:
 - a) high chemical reactivity resulting from a significantly increased specific surface area compared to microcrystalline materials [8, 13, 41, 43],
 - b) increased resistance to corrosion [8, 13],
 - c) different chemical and phase compositions [8, 9, 30, 33, 42],
 - d) high susceptibility to aggregation (chemical bonding of the particles) [30, 42, 43];
- physical, including:
 - a) very small size; ability to miniaturize complex IT systems [8, 9, 42, 65],
 - b) relatively low weight [3, 8, 9, 17],
 - c) changes in crystalline structure (specific structures are stable in nanoscale only) [42, 43],
 - d) high susceptibility to agglomeration (physical particle bonding due to van der Waals forces) [3, 8, 9],
 - e) high surface area to volume ratio resulting in strong sorptive properties (adsorption and absorption) with an increase in catalytic activity of nanomaterials [20, 42, 51],
 - f) significantly lower material melting point [9, 30, 69],
 - g) changes in physical properties including density, solubility and surface tension [8, 9, 30],
 - h) unique optical properties of semiconductors and metals for use in optoelectronics [65],
 - i) changes in spectroscopic properties [49],
 - j) catalytic properties [40, 51],
 - k) good conductivity and superconductivity [10, 13, 61, 65],
 - l) magnetic properties [3, 8-10, 70],
 - m) diffusivity [3, 42, 43],

- n) ability to store energy, e.g. dihydrogen (H_2) can be stored between graphene layers (6-7 nm) at room temperature (a standard qualifier for practical applications is $62 \text{ kg } H_2/m^3$) [48, 50];
- mechanical, including:
 - a) hardness [42, 69],
 - b) abrasion resistance [42, 69],
 - c) slip properties [42, 69],
 - d) creep resistance; for high temperature ceramic structural materials e.g. silicon nitride (Si_3N_4 – good insulator), silicon carbide (SiC – high hardness) which may increase by an order of magnitude for a material in the nanoscale [30, 42, 43],
 - e) mechanical properties including friction, ductility and flexibility [69],
 - f) superelasticity resulting from a decrease in the size of grains of intermetallic cluster bonds to several nanometres [17, 42, 43, 69],
 - g) materials with extremely high resistance to breaking including nanotubes (NTs) [52],
 - h) metal alloys, obtained using mechanical methods which can feature chemical and phase compositions, that cannot be achieved using conventional methods and showing significantly higher mechanical strength and corrosion resistance [69],
 - i) reduced brittle fracture appearance transition temperature [33, 42, 43, 69];
- biological, including:
 - a) good antibacterial and sterilising properties [71, 72],
 - b) easy crossing of biological borders [71],
 - c) ability to use nanocrystalline solids with a dendrite structure as gene carriers (e.g. TiO_2) [73],
 - d) source of oxidative stress; nanoparticles show a similar mode of action to asbestos fibres [73, 74],
 - e) a wide effective range in relation to size and reactivity [8, 9, 14].

Nanotechnology provides innovative methods for obtaining products and nanoobjects with lower energy expenditure and with lower quantities of raw materials required [3, 8-10, 30, 33, 42, 43]. Products containing nanomaterials are more durable, lighter and are more aesthetic [8, 9, 13, 30, 33, 42].

One of the disadvantages of nanomaterials is their metastable nature, in particular at increased temperature with reduced free enthalpy, leading to grain growth [30, 43].

5. Nanomaterial fabrication methods

Numerous studies on new methods of nanomaterials fabrication aim to develop methods which meet a series of strict requirements. The fabrication procedure must be:

- cost-efficient,
- relatively fast,
- easy to replicate,
- provide products featuring stability and size close to the specifications, and
- use low toxicity solvents, reactants and synthesis products [8-10, 22, 30, 75].

Currently developed nanostructure fabrication methods can be based on [3, 8-10, 30, 33, 44, 75]:

- size reduction of solid macro and micro materials to nanoparticles with a specific nanosize, by grinding and milling in ball mills, cutting or lithography, also referred to as the top-down method [8, 30, 42, 44, 75],
- spontaneous bonding of single atoms into larger structures (atom by atom) as a result of the controlled gradual growth of nanocrystallites and further aggregation until a suitable nanoparticle size is obtained, also referred to as the bottom-bottom method [42, 44, 75, 76],
- spontaneous growth of nanocrystallites and atoms into larger aggregates, agglomerates and clusters also referred to as the bottom-up method [22, 43, 44, 75, 77-80].

The bottom-up approach allows nanomaterials with a reduced number of defects and a narrower particle size distribution and particle morphology to be obtained. The most commonly used methods include:

- coprecipitation of products in the nanoscale in solutions, precipitation of the required compound

- as a shell [30, 41, 44],
- applying reactants layer by layer using a Langmuir-Blodgett ultra-thin film technique, commonly used in molecular electronics [8, 42, 44],
- decomposition of organic precursors [22, 78],
- synthesis in hydrothermal conditions [3, 8, 42, 43],
- hydrolysis of reagents and further condensation, e.g. Stöber sol-gel method [33, 76],
- surface atom exchange by redox reaction [10, 30, 33],
- vacuum deposition [9, 30, 43],
- condensation in liquid or gas phase, e.g. spray-drying [17, 30, 33],
- digestion of a surface layer of a specific alloy (e.g. Raney nickel) [20, 42, 81],
- evaporation of metals in an inert gas atmosphere at the pressure of 0.1 MPa using plasma (DC arc plasma method) [17, 80, 82],
- electrochemical redox processes, anodization [17, 32, 80],
- ultrasonic mixing [9, 13, 22, 41, 42],
- burning [30, 42, 80],
- sweeping [43, 80],
- template [8, 30, 43].

Nanomaterials and nanoobjects are usually fabricated on a laboratory scale which means that the processes are expensive and take longer to complete and often involve issues with repeatability and reproducibility. Access to the latest innovative solutions can also be limited, as they are of interest to the defense industry [11].

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