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Review article

DIATOMACEOUS EARTH: A LITERATURE REVIEW

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Abstract

Diatomaceous earth represents a very important industrial mineral with very diverse use and a material with high commercial value. The diverse use and value are incumbent to its nature. The chemical nature of the frustules, the high purity, the morphology, physical characteristics such as very high porosity, chemical inertness and other properties make this industrial mineral very useful as filtration media, abrasive material, additive in the cement industry, starting material for bricks and ceramics, insulation material, etc. This review paper focuses on the most significant uses of the diatomaceous earth.

Keywords: diatomaceous earth, diatomite, kieselguhr, silicate, frustules.

1. Introduction

Diatomaceous earth, also known as diatomite, are common sedimentary siliceous rocks of biogenic origin, siliceous microfossils in the form of diatom frustules, radiolarian cells, silicoflagellate skeletons and sponge spicules. The main component of these microfossils is silica (opal-A), with variable presence of quartz, feldspars, carbonate, clay minerals and organic matter. The shape, size as well as the symmetry of the frustules is of significance for the taxonomy and application. [1-14]

The diatomaceous earth possesses a unique combination of physical and chemical characteristics such as: high porosity, small particle-size, high permeability, chemical inertness, low thermal conductivity, low specific gravity, high absorptivity. [15-16] In addition to the secondary components such as alumina (Al2O3), iron oxide (Fe2O3), lime (CaO), magnesia (MgO), and other minor constituents, amorphous silica (SiO2.nH2O) is major chemical constituent of diatomaceous earth. Diatomite's are found in various colors, white, yellowish, gray, light gray, dark gray, even brownish gray, depending on the level of impurities and chemical composition. [17-18]

Information about the use of diatomites to reduce the weight of structures came from ancient times, and the modern practice of their industrial use is a little over 100 years old. On the initiative of the Swedish chemist and Russian entrepreneur Alfred Nobel in the 1860s, the development of diatomite deposits near Hanover (Germany) started to use for the adsorption of nitroglycerin in the production of diatomite. A decade and a half later, in the United States, diatomaceous earth is used to purify sugarcane syrup. A further increase in the consumption of diatomite was associated with the introduction into the practice of calcination, continuous calcination, calcination with flux and other progressive processing methods. The production of filter materials has increased: In the USA in 1965 for this purpose, 44% of the mined diatomites were consumed, in 1982. - 67%. In Denmark, clayey diatomite is used mainly in the production of lightweight fired bricks. In the USSR, the bulk of diatomites were used in the production of cement (70%), bench materials (15.6%), porous aggregates (5.1%), and thermal insulation (6%); the share of production of natural mineral sorbents accounted for 3.3% of the extracted raw materials. A significant proportion of siliceous rocks (up to a quarter of the total) is used for the production of heat-insulating and building materials. Diatomites and tripoli are widely used to produce various heat-insulating materials and products - bricks, segments, shells, asbesurite, vulcanite, foam diatomite products, etc. Their natural powders are a good bulk heat insulator. The main criterion for assessing the quality of raw materials is the bulk density and the associated thermal conductivity index. The popularity of using diatomite for building materials is due to the lax quality conditions of raw materials - in the production of lightweight building bricks, the content of clay material up to 40% and sandy material up to 15% is allowed.

Fig 1. Diatomite: (a) Raw, (b) Calcined under oxygen, and (c) Calcined under argon flow [13]

Each of the thousands of diatom species studied so far have their own shape and size, ranging from 2 microns to 2 mm [19], Have complex outer shells of silica particles. Since the late 1980s. all over the world to determine new areas of application of diatomites of the main industrially significant deposits of the world (in the USA, China, Denmark, etc.), detailed microscopic studies, analyzes of the taxonomic composition of diatoms, identification of rock-forming species and the main morphometric parameters of their valves were carried out. For Russian science, in time, this period coincided with the well-known difficulties of the 1990s, as a result of which these studies are being carried out only now. Due to the unique three-dimensional structure, diatom valves are now being comprehensively investigated to determine possible uses [20-24]. Brand new areas include composite materials design, photocopying, photonics, molecular separation and biosensorics. A number of studies are also devoted to the study of the physicochemical characteristics of individual parts of the valves in the framework of improving technologies for deep chemical processing of mineral raw materials [25].

The abovementioned characteristics of this industrial mineral make this material suitable for use in a wide range of applications. This review paper summarizes the most significant uses of the diatomaceous earth in various fields, not limited to food industry, pharmaceutical industry, construction industry, use in nano-technology and so on.

2. Diatomaceous Earth - Characterization

Diatomites (from the Greek - *diatome* - cut in two, in the shape of a diatom shell, folded in two halves in the form of a box and a lid) are soft light finely porous rocks, loose or loosely bound, composed mainly of the smallest opal shells (or their remnants) of diatoms, having a size from 0.001 to 0.4 mm. Their bulk density in a piece usually does not exceed one and in the best differences it is 0.5-0.7 and even 0.25-0.3 g/cm^3, density 2.0-2.3 g/cm^3, while the porosity reaches 75-90%. The color of diatomites come in white, yellow, dark and brownish gray, the strength does not exceed 3 MPa [26].
In terms of chemical composition there are major, secondary and minor constituents in diatomite. The major component as always is silicon dioxide (silica), whereas Al_2O_3, CaO and Fe_2O_3 are considered as secondary, while as minor components are Na_2O, SO_3, TiO_2, MnO_2 and other components [27-30]. The content of the major component as well as the secondary and other minor component varies and it's different in every deposit due to its nature of formation and the environment. Besides the major, the secondary and the minor elements, present in diatomite could be various heavy metals as impurities as well as rare elements. The economic importance of certain diatomite deposits is determined based on the ratios of these various components. The higher the amount of silica present in diatomite the higher its quality from the economical perspective. [26, 31].
The amorphous nature of silica present in diatomite implies an abundance of silanol and siloxan bridges on its surface [32]. FTIR analysis have confirmed the presence of characteristic bands for silica around 3600 cm^{-1}, that could be as result of the absorbed water on its surface. A very broad and strong band around 1100 cm^{-1} is related to the Si-O-Si asymmetric vibrations, while the bat at around 800 cm^{-1} is as results of the Si-O-Si stretching vibrations [33-34].

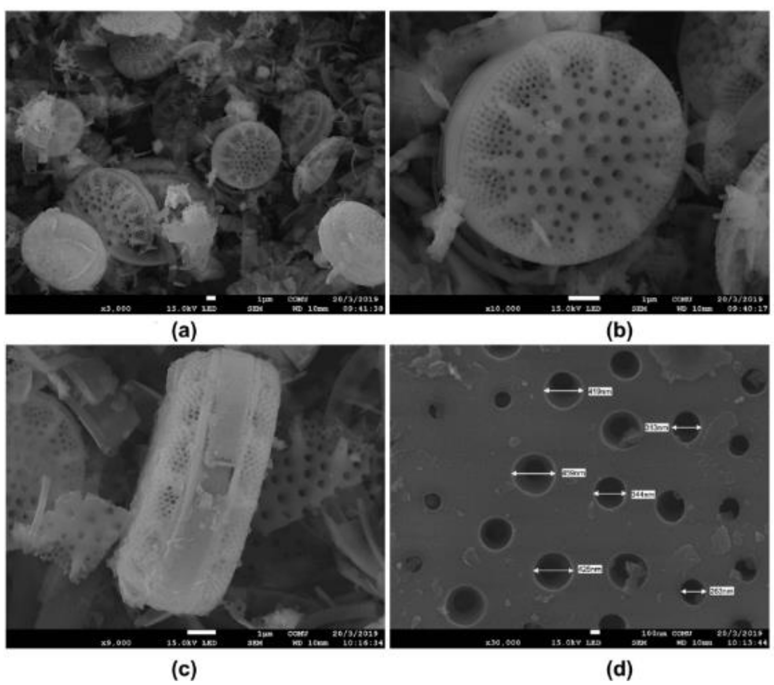

Fig 2. SEM of raw natural diatomite. (a) Several valves of Tertiarius jurijliiand probably one frustule of T. mariovensis in right bottom corner, (b) a whole valve of T. jurijlii, (c) T. jurijliigirdle view, and (d) Areolae in the central area of T. Jurijlii [29].

The crystalline phases found in diatomite are usually present as minor constituents, and they typically include quartz, calcite, kaolinite, feldspar, cristobalite and mica [9, 26] It has been observed that upon calcination the clay minerals are entirely removed at elevated temperatures (1100 C) the entire opal underwent total solid–

solid phase transformation to cristobalite [13]. Moreover, the temperature interval 1,100–1,200°C depicted the formation of mullite [35-37].
Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) results of diatomite show its biogenic origin. Various frustules and/or entire skeletal shapes of diatoms algae ranging from 5 to 15μm were observed. SEM morphology of the diatomite indicates the presence of well-preserved shapes of the diatom frustules. The existence of other forms is also evident, which are in all probability as a result of the clay component found in the material. The size of the pores ranges between 150 and 600 nm in diameter [29].

3. Diatomaceous Earth - Filtration

The diatomaceous earth has been used in many beverage as well as food applications for over 85 years and was utilized especially in the filtration of potable water during WWII. Ever since, it has been utilized to produce low-cost drinking water [38]. There are several hundreds of diatomaceous earth providers in the online market and the vast majority around 60% of the diatomaceous earth production in the US is used for filtration purposes [39]. The use of diatomaceous earth use is not limited for beer, drinking water, oil, liquors, and swimming pool filtration, and could also be used in heated petroleum desalting [40].
The filtration with Diatomaceous earth (precoat filtration) is achieved with the particle removal by physically straining the solid material out of the water. The layer of the diatomaceous earth is about 3 mm thick, while the water pathways are very small and large in number that thus retaining even the very fine particles. Such diatomaceous earth filters are effective in removing algae, iron, manganese, Giardia cysts, coliform bacteria, asbestos and turbidity. Diatomaceous earth filters have low initial cost compared to the conventional rapid sand filtration systems, and are most suitable for water supplies with low amounts of suspended solids, where influent is low in turbidity and bacterial counts. Water supplies presently receiving just chlorination may consider using diatomaceous earth to meet the filtration requirements [41].
Considering the impurities found in diatomaceous earth, França et al. [42] successfully beneficiated the Brazilian diatomite with very good efficiency, while removing the clay, dewatering and calcination of the diatomite. From the tests conducted, the optimum filtration rates occurred at a temperature of 900°C, with flux addition of 3% (w/w) and retention time of 47 minutes. The author concluded that under these conditions, the calcined diatimote showed similar properties to the commercial products being used as filter aids.
Ediz et al. concluded that the calcined diatomite can be successfully utilized as material for beer filtration however upon suitable arrangement of the particle size distribution, in manner that the highest possible flow rates and clarity of the filtrate are obtained. After the turbidity test results, the calcined diatomite was found to be quite comparable to the other commercial filter aids used and the filtration performance of the diatomite can further be improved with a suitable particle size distribution [43]. Moreover, the natural diatomaceous earth can be used combined with a synthetic polymer (polyaluminum chloride - PAC, polyferric sulfate - PFS and polyacrylamide) for the purposes of large-scale filtration water stations, thus initiating a flocculation-coagulation step in which suspended particles such as viruses, algae, clay, inorganic and organic pollutants will flocculate on the surface or at the bottom for easier collection/removal [44].

4. Diatomaceous Earth For Removal of Heavy Metals

The problem of many objects of industrial landfills is their territorial proximity to hydrographic networks, which entails the need to create permeable filtration barriers that can prevent the migration of heavy metals and other pollutants into the environment. One of the main requirements for filtration barriers is effective sorption of various pollutants. Diatomaceous earths are widely used as an effective sorbent in relation to heavy metals. Therefore, the study of this direction is of not only scientific but also applied interest. In addition to

the sorption of heavy metals, diatomaceous earths and their modified varieties are also used for the sorption of organic pollutants and radionuclides [45].
A distinctive feature of diatomite is that it consists of remnants of diatomaceous algae of siliceous composition, which have a high specific surface area due to their very fine average particle size and micro- and macroporosity. Speaking about the sorption capacity of diatomite and other siliceous rocks, it is worth considering separately the opal-cristobalite phase, the main rock-forming component and mineral impurities. Since the surface of opal particles has no charge, the main mechanism of sorption is physical adsorption on the surface of particles and inside pores, which is directly related to the specific surface area. On the other hand, there are works that admit the presence of bonds with silanol (SiOH) groups [46].
As for the impurity minerals, in most cases they are represented by clay minerals - smectite, illite and kaolinite. Even a small admixture of clay minerals can significantly increase the sorption activity of the rock. In this case, ion-exchange is the main sorption mechanism. Thus, diatomaceous rocks combine several mechanisms of sorption - ion exchange and physical adsorption, which significantly expands the efficiency of diatomaceous earths in relation to various heavy metals and other pollutants. As for the pollutants themselves, diatomaceous earths can be effectively used for the purification of Al, Ba, Cd, Cr, Cu, Fe, Pd, Mn, Ni, Zn, as well as Cs, U, oil products and other organic pollutants [47-50].
Both natural and modified DE can be used to remove various contaminants from drinking water efficiently including heavy metals, such as Pb2+, Cu2+, Cd2+, Zn2+, Cr+3 [51-53], organic dyes [54], and harmful microorganisms, as well as some kinds of viruses through depth filtration techniques [55]. The adsorption of Cr(VI) ions onto a clayey diatomite was shown to occur very efficiently with 96.7%. During the experiments the effect of operating parameters, such as amount of adsorbent (0.5–5.5 g/l), initial adsorbate concentration (0.3, 0.4, 0.5, and 0.6 mg/l), and time of adsorption (5–180 min) at pH of the solution 3, was examined. It was found that 2.5 g/l is optimal adsorbent dosage for maximal removal of Cr(VI) ions [56-57].

5. Diatomaceous Earth – Modification and Composites

Gencel et al. [58] used diatomite and polypropylene fiber thus producing gypsum composites. The results showed that the compressive strength of the composites increases while depending on the increment in diatomite content in the mixture.
The preparation of thermal energy storage properties of polyethylene glycol (PEG)/diatomite composite as a novel form-stable composite phase change material (PCM) was performed by [59]. The composite material was prepared by incorporating PEG in the pores of diatomite. The PEG could be retained by 50 wt% into pores of the diatomite without the leakage of melted PEG from the composite. Thermal properties of the obtained composite PCM were analyzed by DSC analysis. DSC results showed that the melting temperature and latent heat of the composite PCM are 27.70 1C and 87.09 J/g, respectively. Thermal cycling test was conducted to determine the thermal reliability of the composite PCM and the results showed that the composite PCM had good thermal reliability and chemical stability. TG analysis showed that the impregnated PEG into the diatomite had good thermal stability. Thermal conductivity of the composite PCM was improved by adding expanded graphite in different mass fractions.
Qi et al. [60] synthesized superabsorbent by solution polymerization of diatomite and partially neutralized acrylic acid. During his work the author investigated the influences of some reaction conditions, such as diatomite content, neutralization degree of acrylic acid, amount of initiator, amount of crosslinking agent, monomer concentration, and the reaction temperature on swelling. The results of swelling rate measurement proved that the diatomite composite superabsorbent had better swelling rate than that of poly(sodium acrylate) prepared in the same conditions.
The TiO2/diatomite composite photocatalyst was obtained by a simple hydrolysis deposition method with the use of titanyl sulfate as precursor. The TiO2 nanoparticles immobilized uniformly onto diatomite. The

TiO_2/diatomite composite showed higher photocatalytic performance for formaldehyde degradation compared to TiO_2 [61].

Zhen et al. synthesized tin oxide/diatomaceous earth composite was via the hydrothermal method without using a template or surfactant. The SnO_2/diatomite composites have a special embedded structure, in which SnO_2 nanoparticles are embedded in the diatomite porous frame. In this study the authors demonstrate a novel strategy for designing a material that can rapidly sense and control humidity and provides insights into the application of the composite in sensing and controlling environmental humidity at room temperature [62].

Agüero et al. manufactured [63] environmentally friendly composites from poly(lactic acid) (PLA) and diatomaceous earth (DE), by extrusion, followed by injection moulding. The data show that the addition of diatomite provides an improved tensile modulus and induces more brittle composites due to stress concentration phenomena. The addition of compatibilizers in PLA-DE positively contributes to improve ductile properties, thus leading to high environmental efficiency materials with balanced mechanical properties.

Marthi and Smith [64] synthesized λ -MnO_2 diatomaceous earth composite adsorbent by using a combination of hydrothermal treatment and solid-state calcination. The synthesized adsorbent was tested with LiCl buffered solution for various batch adsorption parameters. Loading of 28–29% λ-MnO_2 on diatomaceous earth showed the best performance for lithium adsorption.

Sha and Chen [65] prepared ammonium polyphosphate (APP)-diatomaceous earth (DE) composite by in situ polymerization, modified with silane coupling agent (KH550) and was applied as filler to prepare flame-retardant paper. Obtained data showed that chemical bonds occurred between APP-10 % diatomite filler and KH550, and APP-10 % diatomite filler had lower water solubility and better crystallization and thermostability after it was modified with KH550. Paper filled with modified APP10 % DE filler had higher LOI value and lower heat release rate and mass loss at the same filler loading, and its charred residue collected after cone calorimeter test was more compact and strong. Surface modification of APP-10 % DE filler with KH550 improves its flame-retardant effect on paper.

6. Diatomaceous Earth - Construction Material

The diatomaceous earth has been proven to be a great additive especially as an insulating material, starting material for construction materials and ceramics. Due to its low density, this makes the diatomaceous earth a useful material in producing lightweight aggregates.

Cong et al. [66] studied the impact of diatomaceous on the properties of asphalt binder and concluded that the storage stability tests indicated that the diatomite modified asphalt is very stable when diatomite content is lower than 20%.

Kashcheev et al. [67] was able to improve the thermal insulation of high-temperature furnaces with the use of diatomaceous earth and proposed a product that is 50% cheaper, superior in thermal conductivity as well as thermal stability by factors of 2, while Aydin and Gul [68] studied the impact of diatomaceous earth as additive on the concrete properties. The authors indicate that the increase of additive ratio results in a decrease on the compressive strength while [69] concluded that according to the test results, it is suggested that diatomite can be used up to 5% as a replacement of Portland cement in production of cement mortar, and thus mortar containing up to 5% diatomaceous earth exhibited comparable results with Turkish Standard requirements.

Muttaqin et al. [70] used two methods to obtain lightweight concrete. The first method was used to produce lightweight aggregate by sintering chunks of diatomaceous earth and afterwards crushing them. The concrete produced from the aggregates with this method resulted in aggregates with properties only to be used as insulating material (non-structural). The second method involved refining the material to make pellets after which it was sintered. Products from the aggregate made using the second method as coarse aggregate and river sand as fine aggregate can be classified as lightweight structural concrete.

Escalera et al. mixed diatomaceous earth with Brazil nut shell ash in different amounts between 0 and 30 wt% and sintered the mixture at temperatures between 750-950 ºC. The addition of nut shell ash into the diatomaceous earth resulted in significant changes of the microstructure upon sintering. The ash addition produced lightweight porous bricks with acceptable strength at lower sintering temperatures. The obtained products were of high porosity (49%), density 1.06 g/cm^3, thermal conductivity 0.20 W/(m K) while the compressive strength was 8.5 MPa [71].

Reka et al. used diatomaceous earth to obtained porous ceramics under low-temperature hydrothermal procedure. Results showed evidence of products, ceramic materials with low bulk density, high porosity and high compressive strength (14.7-19.4 MPa). During the hydrothermal reaction, the formation of calcium silicate hydrates was evidenced which has positive effect on the compressive strength as well as the porosity of the hydrothermally obtained products [72], while Akhtar et al. [73] fabricated porous ceramic monoliths by dry pressing diatomite powder and by conventional sintering. The sintering behavior of the diatomite powder bodies at various sintering temperatures suggests that diatomite monoliths with a high porosity and strength can be prepared at 1000 °C. The density of the sintered monoliths increases strongly at temperatures 1200 °C, while the compressive strength of the sintered monoliths increases accordingly with the sintering temperature. Keeping the sintering temperature around 1000 °C results in highly porous materials that also display a high compressive strength.

Galotta et al. [74] densified the diatomaceous earth by cold sintering at room temperature and at 150°C under various pressures (100, 200, and 300MPa) while using different NaOH water solutions (0–3M). The relative density of cold sintered diatomite resulted as high as 90%, a condition that can be achieved by conventional firing only at 1200– 1300°C. The cold sintered products maintain the same mineralogical composition of the initial material, while during conventional firing with the formation of cristobalite and mullite, while the bending strength of cold sintered artifacts can exceed 40 MPa and increases to 80 MPa after post-annealing at 800°C, such mechanical strength is much larger than that of conventionally pressed samples sintered at 800°C, which is only around 1 MPa.

7. Diatomaceous Earth - Other Applications

The diatomite application is extremely diverse and it should be noted that it continues to expand, and the structure of application is becoming more diversified. Diatomite is considered as the one of the safest and most effective naturally occurring insecticides [75]. It has a physical mode of action since it kills insects by desiccation. The theory of the diatomite the insecticidal action mechanism was developed as early as 1931 when Zacher and Kunike described the “Zacher effect” [76].

In Western Siberia, where the largest deposits of hydrocarbon raw materials are localized together with deposits of diatomites and opoka [77-79]. This conFig uration created prospects for the use of diatomites and materials based on them for the needs of the oil and gas industry. Despite the relaxation of this topic in recent decades, an extensive theoretical base has been accumulated on the possibilities of their application. The results of various studies presented in domestic and foreign literature indicate the possibility of using diatomites, tripoli and flasks in the reclamation of contaminated soils and water bodies, in the production of chemically resistant thermal insulation materials, proppants, lightweight grouting solutions, in the dehydration and desalination of oil, and natural gas drainage.

The introduction of diatomite as a mineral additive in cement slurries allows a significant decrease in their density. In the 1980 investigated the possibilities of using Novy Urengoy diatomites and diatomaceous clays for preparing lightweight cement slurries in deep oil exploration drilling [80-81]. The results of these studies showed that the addition of diatomite powder to the solution in an amount of 20 to 40% to the mass of dry cement significantly improves spreadability, reduces fluid loss, water separation, and shortens the setting time. The resulting cement stone has a strength of at least 1.96 MPa in bending, 5.08 MPa in compression, as well as low gas permeability. Also, lightweight grouting slurries can be successfully used for fixing sidetracks in problem intervals of water flows, abnormally low reservoir pressure and intervals of high-water-cut production from oil and gas wells [82].

In the chemical industry, diatomites and flasks are raw materials directly for the production of liquid glass (sodium silicate), which is widely used in various sectors of the national economy - construction (for cementation of soils, tunneling of various hydraulic structures) and soap industry, as an adhesive material, for impregnating coatings highways, as regulators of the hardening of binders in the production of cellular concrete [83], etc. The main indicator of the quality of raw materials is the content of amorphous (soluble) silica.

In comparatively smaller amounts, diatomaceous powders (natural and refined) are used as fillers in the paint and varnish, rubber, paper industry, in the production of plastics, etc. Moreover, they play the role of not only an inert substance. With the introduction of diatomite powder, the properties of the product itself change. In the paint and varnish industry, diatomites are among the fillers that increase the strength of the film, the viscosity and fire resistance of paints, and the resistance of films to friction [84]. The introduction of diatomite into polymer compositions increases their fire resistance with a simultaneous improvement in mechanical properties due to the reinforcement with macroporous valves of diatoms, which are, in most cases, openwork two-dimensional siliceous structures [85-86]. The result is achieved with the introduction of 1-2% diatomite, compared with 10% for vermiculite or phlogopite. The results obtained indicate the feasibility of developing fire-retardant compositions based on diatomite.

Due to the high oil absorption, diatomaceous earth is of little use mainly in the production of ink, aniline, alizarin [87], rubber fillers [88]. They easily mix with rubber, approaching kaolin in properties (and sometimes surpassing them), have a relatively low weight. Golstman [89] and Yatsenko [90] utilized diatomite to produce foam glass materials by hydrate mechanism.

In addition to the above-mentioned applications, diatomite has been widely studied as a precursor and template for a range of materials in the energy harvesting and storage industry [91]. Diatomite is considered a potential game changer as far as future energy applications are concerned. In the past decade, nanostructured materials such as; Si, Mg_2Si, SiO_2/C, and TiO_2 have been synthesized from diatomite via chemical reduction reactions and templating for applications in batteries, thermoelectrics, supercapacitors and photocatalysis. Considering the early successes reported to date [91,92], diatomite could attract further attention in other energy related applications.

8. Conclusions

Diatomaceous earth is widely used in all spheres of life and industrial production, which is primarily due to its unique properties and composition. The characteristic properties of diatomite include low bulk density, high thermal stability and specific surface area, the presence of microporosity, sorption properties, as well as the presence of amorphous silica. In addition to the main areas, such as the production of refractory materials, fillers in concrete and asphalt, as well as the sorption of petroleum products, there are many other areas where diatomaceous earths can be used as an effective and inexpensive material.

In the construction sector, in addition to the production of lightweight fillers, diatomite is used as sound and heat insulating materials. Diatomite-based foam glass granules and blocks are highly resistant to moisture, low in weight and cost effective.

A fairly large volume of diatomaceous earth granules is used for producing cat litter. Due to their efficiency and low price, they are popular along with clay fillers. Some varieties of diatomaceous earth, with a low content of crystalline impurities, are used in the abrasive industry - soft polishing powders and pastes for bronze, aluminum, as well as marble and glass.

In the food industry, diatomite is used in the purification of vegetable oils, filtration of sugar, syrups, wine and beer. In medicine - for insulin filtration, as well as a sorbent for food poisoning, detox therapy and drag delivery

systems. In general, diatomaceous earth has a beneficial effect on the human body when consumed as a food additive, but for this, only a purified diatomite is used.
In the chemical industry, diatomite is used as a catalyst, desiccant, dusting agent, filler for rubber, varnishes and paints. In the oil and gas industry it is used as a gas drier and oil desalination agent.
In agriculture diatomite is used as a carrying agent for fertilizers and insecticides, and an anti-caking agent. Diatomites are also used as supplementary feeding for livestock - due to the presence of useful trace elements in its composition and the sorption of mold fungi from the feed, animals gain weight faster and get sick less. In the last century, diatomite was used as an agent for killing fleas, bedbugs and other pests that live indoors and are carried by pets. In addition, diatomaceous earth is a good remedy against cockroaches and garden ants in the house.

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