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Small particles, big changes; synthesis, characterization of nanomaterials and an overview of their application areas

Ebru Halvaci^a, Ozge Ozdemir^b, Mucella Kaya^c, Yuksel Elif Serin^d, Gamze Tekkanat^e, Teslime Kozak^f, Aysenur Aygun^g*

^aSen Research Group, Department of Biochemistry, Dumlupinar University, Kutahya, Türkiye, ORCID: 0009-0003-6062-7622 ^bSen Research Group, Department of Biochemistry, Dumlupinar University, Kutahya, Türkiye, ORCID: 0009-0008-3821-5126 ^cSen Research Group, Department of Biochemistry, Dumlupinar University, Kutahya, Türkiye, ORCID: 0009-0003-4522-7153 ^dSen Research Group, Department of Biochemistry, Dumlupinar University, Kutahya, Türkiye, ORCID: 0009-0005-2989-5949 ^eSen Research Group, Department of Biochemistry, Dumlupinar University, Kutahya, Türkiye, ORCID: 0009-0009-0966-1044 ^fSen Research Group, Department of Biochemistry, Dumlupinar University, Kutahya, Türkiye, ORCID: 0009-0006-9446-8449 ^gSen Research Group, Department of Biochemistry, Dumlupinar University, Kutahya, Türkiye, ORCID: 0009-0006-9446-8449

Abstract

In the age of advanced technology, nanomaterials, their chemical and physical properties, characterization methods and synthesis methods have become very interesting in the scientific world. Considering the size, shape, and synthesis conditions of nanomaterials, and physical structure, the related materials are synthesized at the nanoscale. Nanomaterials have electrical, magnetic, optical, mechanical, and catalytic properties superior to micro materials and have the potential to create new and advanced products. For this reason, nanomaterials are widely used in the healthcare sector, textile industry, automotive technology, energy systems, science, and education. In general, this review covers the synthesis of nanomaterials, characterization methods, and their approaches to application today. In particular, nanomaterials have been obtained from top to bottom or from bottom to top with different specific approaches, and the structures of nanomaterials have been elucidated by explaining the relevant characterization methods. Finally, by giving an overview of the applications of the synthesized and

^{*} Corresponding author. Tel.: 0 274 443 3346 *E-mail address:* fatih.sen@dpu.edu.tr

characterized materials in daily life, the study was completed with the achievements of the nanoscale world. © 2023 DPU All rights reserved. Keywords: Characterization Methods; Nanomaterials; Nanobiotechnology; Nano world.

1. Introduction

The term nanometer (nm) is one billionth of a meter (10^{-9}) , and a single strand of human hair is 60,000 nanometers. For example, it can be assumed that the size of atoms is about 0.1 nanometers [1]-[3]. The term nano was first used in the scientific world by Richard Feynman in 1914. American physicist and Nobel Prize winner Richard Feynman was invited to the annual meeting of the California Institute of Technology of the American Physical Society in 1956 with a speech entitled "There is a Lot of Room at the Bottom" [4]. In this speech, he took his place in the scientific world with the definition of the specific concept of nanotechnology and the related studies he has done. The American Journal of Physics came up with the following concept immediately after this speech: "Why can't we write the entire 24-volume Encyclopedia Britannica on one pin?". Along with Richard Feynman's speech and related conclusions, he emphasized that the laws of nature do not limit studies in the field of nanotechnology at the atomic and molecular level, the main deficiency is in terms of relevant equipment and technical knowledge. As can be deduced from this conversation, nanostructures help to illuminate the unknowns of the scientific world with scientific and technological approaches. For the general definition of nanotechnology, in addition to the aforementioned work of Richard Feynman, in 1974, Norio Taniguchi became the second person to use the concept of nanotechnology and said the following sentences: "Nanotechnology mainly consists of the processing, decomposition, combination and deformation of materials by an atom or a molecule."[5]. Nanotechnology deals with the physical, chemical, and biological properties of structures and atoms at the nanoscale, as well as materials and systems that are renewed. This branch of science, which is groundbreaking in various fields today, can also be characterized as the new industrial revolution or the age of advanced technology. In this context, nanotechnology has become a branch of science that studies and researches the behaviour of matter in dimensions between 1-100 nm. With another approach, if the size of a usable nanostructure is considered to be 1-100 nanometers, it can be seen that the field of study of nanotechnology is atoms and structures at the molecular level. Together with this approach, the synthesis, characterization, and functionalization of nanoscale materials in a controlled manner, making them usable at the nanoscale, is the goal of nanoscience and nanobiotechnology [6]. Small research carried out at the micro level in the field of nanotechnology leads to radical innovations in various fields. In addition, the differences in nanoscale structures are not only related to the small size but also related to the fact that they reveal different physical properties at small sizes. The best example of this is that the quantum properties become obvious as the size of nanomaterials decreases, or the geometric arrangement of atoms affects the physical properties of matter. Carbon-based materials, bismuth crystals, and gold can be cited as a good example of this [7]. While the bismuth crystal is a macroscopic metal group element, it shows a semiconductor property in the form of a nanowire [8]. Gold, on the other hand, appears yellow in macroscopic size, while it appears red when examined at the nanoscale. In other words, it has been observed that materials with different geometric, electronic, and optical properties, although they are composed of the same atoms, have different characteristics and structural properties in nanoscale structures [9], [10]. However, reducing the size of the material by reducing its size reveals a different characteristic feature. The size of material at the nanoscale is related to the freedom of movement of the free electrons it holds in its structure in Oct (x, y, and z-axis). If the free electrons are moving in all three directions, then the material is called a "3D" (three-dimensional) structure. The solid materials we encounter in our daily lives are examples of 3D structures [11]. If free electrons can move in two directions, these are materials with a "2D" (two-dimensional) structure. 2D materials are observed in layered structures, one layer may consist of a single kind of atom, while the other layer may consist of another kind of atom. To give an example, group 4A elements such as Silicon (Si), and Germanium (Ge) are layered and belong to the 2D material class [12]. Materials whose free electrons have the property of moving only in one direction are called 1D (one-dimensional) structures. Carbon nanotubes, metal oxide nanotubes, or nanowire structures are examples of 1D materials [13]. On the other hand, 0D (zero dimension) is a nanostructure containing free electrons, but in the form of nanoball and nano-dispersion, which can isolate each other. For example, core nanoparticles, hollow nanoparticles, fullerenes, and quantum dots are zerodimensional materials [14]–[16]. Although 0D materials do not seem to have a specific electronic function, they are indispensable dimensions in various fields, including nanoscience and quantum computing applications. To mention this, materials with different nano-dimensions can show various structural and characteristic properties. The 1D bismuth crystal shows three different structures and conformational properties. These are rod structure, tube structure, and atomic sequence. The inner part of the rod form of bismuth crystalline shows a crystal structureproperty. The tube form is a hollow sphere, and the bismuth crystal, which is formed in the form of an atomic array, consists of atoms attached to the surface. All of these three-dimensional structures are evaluated as nanowires. The synthesis of these materials with different sizes has developed high-activity catalysts, optical applications, advanced technological devices, surfactants, drug carriers, superconductors, pharmacological drugs, and therapeutic products along with exhibiting extraordinary properties [17], [18]. That is why nanotechnology has led to the awarding of Nobel Prizes in various branches of science in the 21st century. At the same time, it has become a multidisciplinary branch of science where basic sciences and applied sciences intersect. In other words, the most obvious advantage of nanotechnology is that by using smaller devices, the performance increases exponentially along with the reduction of the material size [19]. Thanks to this, nanotechnology has a fairly wide field of study for basic disciplines such as engineering, materials science, chemistry, biochemistry, physics, and medicine [10], [20]-[25].

2. Synthesis of Nanomaterials

It is possible to synthesize and characterize nanomaterials by various methods. There are 2 approaches to the synthesis of nanomaterials. These approaches are top-down and bottom-up. The first group, called top-down, treats the material as a whole and starts processing, at the end of the process, the material is divided into small particles. This process allows the material to reduce the structural shapes of microscopic elements to the size of nanometers using chemical etching or surface shaping techniques. In top-down approaches; basic processes such as mechanical milling, electrospinning, lithography, spraying, arc discharge method, and laser ablation method are used. Bottom-up approaches explain the synthesizability of atoms, molecules, or compounds existing in the structure of the material as a result of dimensional growth and clustering as a result of chemical reactions. For this reason, nanomaterials of atomic or molecular size are brought together in clusters in such a way that organic decay, multi-molecular structures, and macro-molecules form structures. Chemical vapor deposition (CVD), solvothermal, and hydrothermal methods, sol-gel method, soft and hard decoupling and reverse micelle method are among the bottom-up methods that hold the nanomaterial together (Figure 1) [26], [27].



Fig. 1. Basic approach scheme for top-down or bottom-up synthesis of nanomaterials [16].

2.1. Top-Down Methods

Mechanical Grinding Method

Mechanical grinding is a cost-effective method used to produce materials at the nanoscale. In this context, the ball grinding system, which is one of the mechanical grinding systems, is the most preferred method. The ball grinding system was developed by John Benjamin in 1970. It is performed by applying intense mechanical energy to the nanomaterial contained in the form of balls and dust particles in the grinding medium. This system is based on the energy transfer resulting from collisions between the Decaying medium and the sample. In the first stage, fine and irregular particles are formed as a result of the dust Decoupling or sliding between the balls. When the second stage is passed, the nanoparticles formed undergo deformation. At the last stage, fractures are observed in the sample that has undergone deformation and broken into pieces. At the end of these stages, the production of nanocomposite materials of various sizes is carried out. Among the advantages of the ball grinding system, it aims to produce a large amount of high-purity materials, increase the Decolonization of slightly soluble substances in water at affordable costs, and give new properties to the products formed. In addition to these advantages, there are also some disadvantages. The long production time, Decontamination of nanoparticles that will form steel balls in the mechanism, and very high energy requirements during material synthesis can be considered among the disadvantages. The materials produced in the ball grinding system synthesize nanomaterials, which provides the opportunity to meet innovative approaches [26], [28].

• Electrospinning Method

The process of producing nanofibers from polymers using electrostatic forces by Formhals in 1934 is called the electrospinning method [29]. With the electrospinning method, an electrical charge is applied to the polymer solution by applying a high potential voltage to the solution. After this step, a polymer gel is formed, which emerges from the fine jet and flows towards the grounded target placed opposite the assembly [30]. During this current, the polymer jet is scattered into very thin fibres, resulting in nano-sized fibres. Since electrospinning, the energy source, and one end of the collector (collector plate) are connected to the ground, it is important to enclose the system in a closed circuit. For this reason, one of the two electrodes in the circuit is placed correctly in the polymer solution, and the other is connected to the opposite collector. The pump placed in the back part of the electrospinning system creates a continuous pressure by pushing the polymer solution in the glass tube through the pipette towards the metal plate. Together with the given current, the polymer solution creates an electrical field in the glass tube. On the surface of the solution, an electrical charge occurs and exerts a force in the opposite direction to this surface tension. The electric current charged to the system can reach up to 30 kV, and when the intensity of the electric field gradually increases, it is observed that the hemispherical liquid in the layout forms a conical shape [31]. This conical shape is known as the Taylor Cone [32], [33]. In this conical way, as soon as the electrical force defeats the surface tension, the charged polymer jet is rapidly directed out of the pattern. As a result of nanoparticles with the same electrical charge repelling each other, the polymer jet is separated into very thin fibres, and collected on a metal plate. While the polymer jet accelerates under the influence of electrical force, the viscous resistance gradually increases. As soon as the viscous resistance is equal to the electrical force, the polymer jet begins to show an unstable distribution [34]. During this movement, even a slight air turbulence causes the material to oscillate. Generally, nanofibers with a diameter of 40-2000 nm (0.04-2 microns) can be produced by the electrospinning method [35]. Parameters such as the concentration of nano-sized fibres, the distance between the capillary tip and the metal collector, and the potential flow Deceleration at the extreme point affect the synthesis of the desired nanomaterial [36]. Polymeric materials produced by this method, carbon-based ceramic nanofibers, compared to other nanotubes, have various advantages in such matters as cost and single-stage material production [37], [38]. In addition, it is often used in the production of nano, and micro-sized medical purposes, ceramic coating, and flexible and fireproof materials based on their size [39]-[41].

• Lithography

Lithography is a printing technique in which painting and writing are combined side by side or intertwined on a flat surface sheet from the past to the present. If this technique is to be adapted to nanomaterial synthesis, it is a convenient, simple technique that improves the nano architecture using a focused light or electron beam [42]. The lithography technique is divided into two main groups in the form of masked lithography and unmasked lithography [43]. Masked lithography covers photolithography, nanoimprint lithography, and soft lithography techniques. Masked lithography nanopatterns perform processing on a large surface area using a specific pattern and template. It covers unmasked lithography, scanning probe lithography, focused ion beam lithography and electron beam lithography [44]. In mask-free lithography, on the other hand, random nano pattern making is performed without the need for any template [45]. the 3D free nano patterns are integrated by a focused ion beam in combination with wet chemical deposition. Lithography is an alternative method that is most often used in printing, pressing, and molding techniques instead of photons and electrons [46].

• Spraying

Spraying is a general method of producing nanomaterials by treating solid surfaces with high-energy particles (plasma or gas) (Figure 2). The spraying process can be carried out in different ways. Flamethrower pyrolysis and

magnetron sputtering are examples of these methods. Magnetron sputtering is a method by which very thin metal coatings are obtained as a result of gas treatment in vacuum chambers emptied under a certain pressure. This method is based on the conversion of solid material into gas by breaking atoms from solid material as a result of high-speed treatment with high-energy ions. The sprayed ions are inert gases and argon gas is usually used as a spray source, while metal surfaces are used as a solid material. As a result of the collision of metal atoms and gas molecules, atoms scattered in the vacuum chamber are scattered. The spraying process allows the material to be atomized and deposited in a substrate to form a layer. Thanks to the inert gas used as a spray source, low-pressure plasma is produced. Another method, flame retardant pyrolysis, has been used in laboratory or industrial areas since twenty years ago to obtain nanoparticles quickly and in a single step. This spraying method involves mechanisms related to the liquid and gas phases of a large number of metal oxide nanoparticles. The metal-containing precursor is dissolved in a flammable carrier solution with the help of oxygen. Evaporation and transformation of the precursor substance are achieved thanks to the heat released into the environment after spray ignition. Nanomaterials produced in gas-to-solid transformations have crystal purity and single-mode size distribution. Since the nanoparticles obtained by this method have high purity, they are preferred in applications. Nanomaterials produced by flame spray pyrolysis are used in many fields such as catalysts, batteries, gas sensors, advanced pigments, biomaterials, food supplements, and nanotoxicology screening studies. In general, the spraying method is used to produce layered or thin nanofilms from nanomaterials. Nanomaterial production is achieved as a result of the collision of the inert gas sprayed at high speed with the metal melt [5], [26], [47].



Fig. 2. Demonstration of the flame spraying method used for the analysis of nanoparticle production stages from metal oxide material. As the ignited gas, a mixture of zirconium n-prop-oxide and ethanol is distributed and ignited with the help of oxygen. Monitoring of the volume of liquid feed (LF) and dispersion gas (DG) and whether the nanoparticles have reached the desired size should be carried out (Reprinted with permission from [48], Copyright WILEY).

• Arc Discharge Method

The arc discharge method used for nanomaterial synthesis and nanoparticle production is mainly performed in an environment where noble gas and reactive components are present. The metal wire on the system essentially creates a current in the form of plasma and collects the material together with the formation of its magnetic field. Dec. For this reason, the electron source in the system creates high-energy sources with plasmas and electron beams in such a way as to form an arc. A fairly high temperature is applied to the reactive components and nanomaterials are synthesized by creating a high current on the metal wire in a short time [49]. In addition, as a by-product during the arc discharge method, a high electric current is needed, so heat is released. A current is applied between two graphite rods positioned at a close distance, one pole of which is the anode, and the other pole is the cathode, in such a way as to create electrical energy. Carbon accumulation occurs at the cathode pole, while graphite rods, which are considered a reference, are used at the anode pole [50], [51]. This synthesis method often produces fullerene, carbon nanotubes, multilayer graphene, and amorphous spherical carbon-based materials [52]. Among the given materials, the arc Decrement method is used most often in the production of fullerene. The production of fullerene is mainly based on the collection of nanomaterials formed on the electrode surfaces of two graphite rods under a certain helium pressure. Since the presence of moisture and oxygen in the environment will prevent the formation of fullerene, the presence of pure helium in the chamber is important. In addition, the arc discharge method is used to obtain graphene efficiently [53], [54]. It affects the nanomaterial formed under the current conditions in graphene synthesis. The nanomaterials that will be formed can be collected on the anode or cathode surface or on both electrode surfaces. In addition, collections can also be observed in the inner chamber, unlike the electrode surfaces [55]. The conditions under which material synthesis takes place in the arc discharge method lead to differences in the forms of the nanomaterial formed. The arc discharge method allows the synthesis of different material groups under changing electrode groups or ambient conditions [26], [47].

• Laser Ablation

Laser ablation synthesis is the process of removing matter from the surface using short and intense laser pulses that hit the target material. The laser beams sent to the target material are first transferred to the electrons contained in the solid, and with this high-energy transfer, the nanoparticles are vaporized. The process of material removal from the target surface is explained by the weakening of bonds due to the absorption of light by the substance. Depending on the intensity of the laser beam incident on the target surface, a plasma cloud is formed with ions, atoms, electrons, or larger particles that break off from the surface. The process of plucking metal atoms from this electron cloud has led to various methods and approaches. The laser ablation technique is a powerful method for processing brittle hard and heat-sensitive substances and for thin film production. Therefore, the efficiency of the ablation process is understood by the ablation rate. The ablation rate is determined based on the average ablation rate per single laser pulse and the thickness of the removed layer. The ablation rate varies depending on parameters such as photon energy, laser flux, beam width at the focal point of light, internal pressure, enthalpy of evaporation, optical processing depth, wavelength, photon repetition rate, pulse energy, pulse duration, energy density, etc. Accordingly, the reduction of the laser wavelength increases the ablation rate. The short wavelength laser beam causes small particles to break off from the target surface easily. The repetition rate is the number of pulses produced by the laser system in one second. Due to the accumulation of thermal energy depending on this period, it causes an incubation effect. The repetition rate increases and decreases in direct proportion to the amount of substance to be extracted from the target surface. Depending on these approaches, the property of the material to be synthesized varies depending on the reactions it will give to laser light. For example, in strong and intense pulsed laser systems, it does not matter whether the material has a metal or dielectric structure since dielectric ionization will occur very quickly. Based on these approaches, the laser ablation process is studied in three main groups: thermal, photochemical, and photophysical. The first method, material ablation in the thermal approach is based on a

heating or evaporation process using laser light. The term photochemical laser ablation, on the other hand, occurs by directly separating molecules, indirectly breaking molecular bonds due to material impurity and stability. Finally, the third method, the photo physical ablation technique, non-thermal approaches determine the ablation intensity of the material. When these methods are also examined, the laser ablation process varies depending on the laser system used and the property of the target substance to which the laser beams are transmitted. The property of the laser system depends on the speed of ablation and the mechanism of its realization. For this reason, it is very important to determine and implement the appropriate experimental stages for the target substance laser system to be selected. Depending on these approaches, the laser ablation process is used in the synthesis of carbon-based nanomaterials, the production of oxide composites, and various nanomaterials synthesis in the ceramics industry [5], [26], [47], [56].

2.2 Bottom-Up Methods

• CVD (Chemical Vapour Deposition)

The CVD (Chemical Vapour Deposition) method is a very old nanomaterial synthesis method. The first practical application can be given as an example of coating the filaments with carbon or metals to make incandescent lamp filaments durable in the 1880s. In the same years, Ludwig Mond and other researchers developed the chemical vapour deposition method (CVD), the carbonyl process, to obtain pure nickel. The method, which has been developed over many years, has been used for the production of high-purity refractory metals such as titanium, tantalum, and zirconium. In addition, it has been a guiding device that has developed in various processes from material synthesis to purification. Among the application areas of CVD, the thin film coating process is used Decently on the surface. In this method, the reaction takes place while the raw materials, which are in the form of gas or vapour, are transported to the compartment called the hot substrate. Reactions occur on or near hot surfaces. Solid products are deposited on the surface as a thin film and various materials are synthesized by this mechanism (Figure 3). If we give an example of these materials; the use of carbides coated with CVD in the industrial field and diamond coatings can be made. CVD is a very good nanoparticle synthesis method in the production of carbon-based nanomaterials, metal deposition, solar cells, microelectronics, and insulating material production [57].



Fig. 3. The working mechanism of the CVD device, the gas inlet outlet, and the compartment where the sample is located [57], [58].

• Solvothermal and Hydrothermal Method

The solvothermal method is a method used for the synthesis of oxidized and non-oxidized crystalline materials. For example, highly porous structures such as zeolite, silicate-based materials, crystalline solids, and nanoparticles are produced by this process. The non-oxidized nanomaterials that can be produced by this method can be given semiconductor structures, borides, carbon nanotubes, phosphides, diamonds, nitrides, carbides, and chalcogenides. In the solvothermal technique, it is a method based on the synthesis of nanomaterials with a solvent, that is, a solvent in a closed container, rising far above boiling points. Hydrothermal method, on the other hand, 19. It has emerged with the branch of geology that started in the middle of the century, studying certain mineral and rock formations, and simulating hydrothermal conditions. In hydrothermal synthesis, the formation of nanomaterials takes place at a temperature higher than room temperature. The reaction system is based on the principle that an aqueous solution reacts with a special sealed container by heating and applying vapour pressure. A substance that is insoluble or slightly soluble in the solvent is dissolved by the hydrothermal method and regains a crystalline structure. The reaction kinetics and crystal growth kinetics of the hydrothermal method occur when reactants dissolve in the hydrothermal environment, ions and molecules also enter the solution by forming a bulk. They are Decoupled according to the temperature differences between the two layers of the boiler at the top and bottom. Ions and molecules contained in groups are adsorbed in the form of vapour. It then decomposes and undergoes desorption. The adsorbed product is in motion at the intermediate level, and the Decayed substances crystallize at the end of the process (figure 4). The same crystals formed show different morphological properties under different hydrothermal conditions. In fact, this method has quite significant advantages over others. With the production of unstable nanomaterials at high temperatures and the method used, minimal material loss is observed. Solvothermal and hydrothermal methods are quite preferred methods in nanomaterial synthesis. The only feature that distinguishes the solvothermal method from the hydrothermal method is the synthesis of nanomaterials in a non-water environment. In addition, it takes place in closed systems in two methods. As for material synthesis, it is used in the production of various geometric structures such as nanowires, nanorods, nanotubes, and nanospheres [59], [60].



Fig. 4. Diagram of typical hydrothermal method equipment [61].

• Sol Gel Method

It is a wet chemical technique used to produce colloidal nanoparticles from the liquid phase. Metal oxides are at the beginning of the nanomaterials produced using this method. The metal oxide synthesis process is completed in several steps At the first stage, the metal oxides are dissolved in water or alcohol, heated by hydrolysis/alcoholises, and converted into a gel consistency by mixing [62]. Then condensation occurs. During the condensation phase, oxo-(M-O-M) or hydroxo-(M-OH-M) bridges are formed. It causes the formation of metal-hydroxo- or metal-oxopolymer in solution. As a result of the condensation process, the solvent viscosity increases and is allowed to age, and porous structures are formed. During the aging stage, the pore structure decreases, and the distances between colloidal particles increase. At the end of the aging process, water and organic solvents are removed from the gel and the drying process takes place. The calcination process is required for the production of nanoparticles. With the calcination process, the formation of dust or biofilm occurs in the material. The product obtained and the influencing factors are the nature of the material taken from the guide, the hydrolysis rate, aging time, pH the amount of water, and the molar ratio of the material in the guide. Advantages such as the cheapness of the material produced by the sol gel method, homogeneous distribution, and low processing temperature facilitate nanomaterial synthesis. Due to the interest in energy conversion and storage applications in the last few years, extensive studies have been conducted on lithium-sodium ion batteries, fuel cells, photocatalytic applications, sol gel syntheses of 2D nanolayers, ceramic and glass production, metal oxide-based aerogels, hierarchically porous monoliths sol gel synthesis [5].

• Soft/Hard Templates

They are simple traditional methods that are widely used in the production of nanoporous materials. The application of soft templating provides an advantage in the development of materials with simple, relatively moderate conditions and various morphologies. A large number of nanoporous materials, block copolymers, flexible organic molecules, and anionic, cationic, and non-ionic surfactants are being produced. In the substance to be synthesized, a nano-sized material is obtained through hydrogen bonds, Van Der Waals interactions, and electrostatic forces. Examples of this are nanomaterials such as mesoporous polymeric carbon nanoparticles, single crystal nanoparticles, porous alumina, and N-doped graphene. Another method, hard stencilling, is also called nano casting. The use of solid materials as templates is the process of filling solid template pores with precursor molecules to obtain nanostructures. In rigid templates, it is desired to maintain the porous structure during the pioneering transformation process and to ensure that the nanostructure produced can be easily removed without deterioration. For this reason, the choice of a rigid template is important. A few of the materials used are; carbon black, carbon nanotubes, colloidal crystals, and silica-based materials. These templating methods take place in three main steps on the synthetic path. The first step is the selection or development of the appropriate original template. Then, the desired precursor is filled into these template pores and converted into an inorganic solid. It is removed with the template for the desired porous copy to be obtained. With the use of these mesoporous templates, nanowires, nanostructured materials with 3D nanostructures- metal oxides, and various nanoparticles are produced [5].

• Reverse Micelle (RM) Method

Reverse micelles are called nanometer-sized surfactant clusters containing water molecules encapsulated in a non-polar solvent [63], [64]. For the first time in 1982, the synthesis of a nano material was performed by Boutonnet et al. using the reverse micellar method [65]. In water, the oil emulsion is hydrophobic, the tails are directed towards the core, which traps the oil droplets in it, resulting in normal micelles. However, the condition of a water emulsion of hydrophilic tips in oil in a core containing water results in the formation of reverse micelles [3]. Reverse micelles

are evenly coated with a single layer of surfactant and in an oil phase, isotropically distributed nano droplet structures are seen [50]. The activity and conformation of enzymes in the reverse micelle method are an example of the formation of droplet structures. In this method, three methods are considered to switch enzymes to the fat phase [66]. The first and simplest of these methods is the injection method. A hydrocarbon solution is prepared for the injection method, and a small amount of concentrated aqueous protein solution is added to the solution. Another second method is the dissolution of proteins in a hydrocarbon micellar solution. The last method is the preferred phase transfer method for proteins that are slightly or not soluble in water at all [67]. However, the reverse micellar method, along with biotechnology and bioorganic approaches, addresses a large number of application areas, including transformations of water-insoluble substrates [68].

3. Characterization of Nanomaterials

The characterization process examines the relationship between the structure, performance, and production of a nano-sized material Decently. Various analytical techniques are used to detect and measure nanoparticles. In these analytical techniques; various parameters such as optical, morphological, electrical, magnetic, physical, and chemical are the decisive elements for material characterization. Nanomaterial characterization studies the size, shape, porous structure, solubility, chemical composition, crystal structures, charge potential and surface area of molecules. SEM (Scanning electron microscope), TEM (Permeable electron microscope), AFM (Atomic force microscope), XRD (X-ray crystallography), UV-VIS (Visible light microscope), FTIR (Fourier transform infrared spectroscopy) devices are used for the characterization of nanomaterials (Scheme 1) [69]. These nanostructures are materials that function in many other fields such as the cosmetics and pharmaceutical industry, catalysis reactions, biomedical applications, chemical sensor systems and medical imaging devices [70]. It is possible to study the characterization of nanomaterials using microscopic, spectroscopic, and spectrophotometric-based devices with various parameters.



Scheme 1. Schematic representation of microscopic, spectroscopic, and spectrophotometric analyses of methods used in the characterization of nanomaterials.

3.1. Microscopy-Based Characterization Techniques

• Scanning Electron Microscopy (SEM)

It is a measurement made using electrons in motion on highly magnified images of nano-sized materials. This system is based on bombarding the sample by focusing it with an arc that produces electrons. It is a microscopic analysis used to image nanoparticles of very small sizes [71]. The Decrement rate of SEM is used in the analysis of organic and inorganic materials at the nanoscale between 300,000-1,000,000. When working with SEM, the sample to be studied first needs to undergo a detailed examination and preparation phase before entering the microscope. First of all, the sample should not exceed 100 nanometers in size. In scanning electron microscopy, only conductive ions are studied. Non-conductive samples are made conductive with special layers such as gold, platinum, or carbon [72]. At the same time, an electrostatic charge occurs when working with conductive ions. Due to the magnetic field, a grounding process must be applied before the samples enter the microscope. After the sample has been examined and passed through the preparation stage, it is glued to the holder with epoxy adhesive or carbon adhesive tapes. A hole is located where the sample was found. Through this hole, an argon beam is sent to the sample, and images are taken by dropping on the material. In these images, grayscale is heavier, and the material can be clearly distinguished. SEM device equipment, maintenance, and workmanship is a device that needs to be taken care of [73]. When SEM is used together with other disciplines, innovative approaches are seen in the imaging of the material and device performance. Scanning electron microscopy gives better results about the composition, elemental analysis, location, and shapes of samples when worked together with an X-ray detector (EDX) [74]. SEM is widely used worldwide, not only in the characterization of nanomaterials but also in in-situ materials engineering. In addition, secondary electron (SE) and backscatter electron (BSE) are being studied together and this technology is also needed in imaging. When working with conductive materials in the SEM device, the secondary electrons (SE) are first thrown to the surface quickly to form an image. This image is visible with a small diameter electron beam. Backscatter, that is, the primary electron beams do not have a good resolution; it does not prevent the determination and information about image concentrations and atomic numbers [75]. As mentioned above, SEM is one of the few devices that is highly preferred and used in many disciplines around the world because it offers more different imaging techniques.

• Transmission Electron Microscopy (TEM)

Permeable electron microscopy is a type of microscope that emerged in the 1990s as a result of demand, requirements, and research. It allows us to study atomic-scale structures and the chemical environment in which the material is located at the nanoscale. Permeable electron microscopy consists of two parts. These are; resin and hardening bases. In these yellow sections, it is first mixed with a toothpick in a petri dish and the sample is added to it. The sample is glued in aluminium foil on small weights with a hot plate and epoxy adhesive is poured on it. After the sample preparation stages, it is possible to take an image easily on the device. TEM is a tool that allows us to get clearer images by interacting with high-energy electron beams thanks to the ability to see the sample at ultra-high resolution [76]. In characterization with permeable electron microscopy, nanomaterial structures such as nano tubes or nano-sized particles, and powders ground by the mechanical method are studied. In TEM analysis, two different types of products are prepared according to the sample to be studied. The first of these is the preparation of nanoparticles with a non-magnetic structure and the other is the preparation of nanoparticles with a magnetic structure using methods and methods suitable for the device. The nanoparticle, which does not have a magnetic structure, is subjected to ultrasonication for five minutes. This process is the process of distributing the material in solution by applying ultrasound at a low temperature. It is very important for this process that the solution used does not clump. Therefore, attention should be paid to the interest of the solution, it should not leave carbon and evaporate when it remains at room temperature for 10-20 minutes [77]. Since the samples are mostly carbon-based,

it is very important that they leave residues in the environment. To determine whether there is a residue, a few drops of the solution should be dropped into a clean glass and evaporated TEM [78]. In addition, if it becomes transparent in water, it means that there is no carbon residue in that sample. When the desired properties are achieved, the flat grid or C perforated grid in the microscope is slowly and horizontally emptied into the beaker with the help of forceps. The grill is dried on filter paper under an infrared lamp. If there is no grid or it is not desired to be used for this process, a substrate with a thickness of 100 nm can be used. In addition, the sample is thinned before it is given to the device, this process allows you to get a clearer and better resolution image of the material from the device. Nanoparticles that have a magnetic structure, i.e. magnetic powders, are more difficult to prepare than powders that do not have a magnetic structure [79]. The reason for this is the agglomeration of magnetic powders, that is, the clustering of materials with larger particle sizes. Although they are easily Decayed by ultrasonication, they come together and cluster together again as soon as sonication stops [80], [81]. Therefore, the sample is placed between the pole parts of the device on the objective lens Dec. Since polarization in the material is an undesirable event, it is recommended to use TEM by going through certain stages for particles that have a magnetic structure. Permeable electron microscopy (TEM) is more often preferred than other types of microscopes because it helps us to elucidate the fibre, nanoparticle, and internal emission structure of many particles and nano-sized materials.

• Atomic Force Microscopy (AFM)

It is studied with atomic force microscopy on various surfaces such as polymer membranes, tissues, cells, proteins, nucleic acids, and functional materials. Morphological and physical properties of high-resolution research, it is possible to characterize various biological and synthetic bio-interfaces, that is, microorganisms or biomolecules are in communication with natural and synthetic structures with the AFM device. There are probes on the atomic force microscope that scan the molecule and allow us to obtain three-dimensional images. The feature that distinguishes these probes from other types of microscopes is that they obtain imaging in both solid and liquid media. The probe surface mimics it by changing the chemistry of the molecule under study. This makes it possible to investigate the mechanical, chemical, conductive, electrostatic, and biological properties of more surface interactions in the environment [82], [83]. Atomic force microscopy is very important because it can map the structures of bio aerosols with high resolution with a signal-to-noise ratio from the microscopic scale to the subnanometer scale [83]. The molecule to be examined in this device goes through a preparation stage. Then, whichever material is being worked with at the tip of the microscope, the adjusted needle and the molecule surface interact in contact. Thus, the desired image can be provided on the device without damaging the molecule. Since the AFM device allows us to study different molecular structures, structures that support this microscopy have been invented to study and investigate in various fields (Scheme 2). The optical detection system of AFM and its derivative Bio-AFM, which allows it to work in aqueous solutions that detect liquid cells, is Bio-AFM. When shaping the sample to be studied, its derivative, which shakes the probe tip to reduce friction and has a dynamic mode, is DM-AFM. It is the FD-AFM that shapes the surface of the biological system based on the force and distance curve. MR-AFM is a type of multi-parameter microscope that maps the contours of the sample being studied by looking at its physical and chemical properties. While drawing the lines of the sample, its derivative, which maps multi-frequency in its physical parameters, is MF-AFM. OPTO-AFM is a microscope that shows better by providing advanced optical imaging for complex images. The AFM derivative, which is mapped at a high speed by accelerating the image 1000 times and showing the biological sample accordingly, is called HS-AFM [83]. The atomic force microscope is mapped according to the concentration and atomic resolution of the sample to be examined. The images are obtained with constant altitude mode and typical atomic resolution, i.e. based on the Pauli principle. Fixed-height AFM images make the sample appear brighter. In addition to the types and variety of microscopes with AFM, the chemical bonds of the atom are observed [84], [85]. This feature distinguishes atomic force microscopy and its derivatives from other microscopes.



Scheme 2. Derivatives of atomic force microscopy in various types and characterization analysis.

3.2. Characterization Techniques Related to X-Radiation

• X-Ray Crystallography (XRD)

XRD is a device that displays diffraction axes that reflect the physicochemical properties of the material when it collides with a wide-ranging and crystalline nanomaterial. The three-dimensional materials that make up the crystal structure are crystalline. Various microscopic and spectroscopic methods have been tried to study and understand the phases of crystalline materials. These methods worked in the characterization of nanomaterials, but the XRD device was invented because the desired result could not be achieved. X-ray crystallography studies the physicochemical properties and crystal structure of materials in detail. The voltage arc formed during the characterization stage provides information on the structure of nanoparticles, electron density, and many other topics [86]. It allows the characterization and research of nanomaterials and nanostructures in this direction. When analysing materials at the nanoscale, powder diffraction X-rays are used. Powder diffraction, also known as the Debye and Scherer method, is one of the methods that require the least material. These methods can be performed quickly, and qualitative analyses easily without damaging pure and multi-component mixtures. If there is a very small amount of samples for nanoparticles or nanomaterials to be studied, it is easier to work with X-ray crystallography than other analysis methods. Therefore, he works with XDR devices in nanomaterial and nanoparticle characterization, nanomaterial extraction, catalysis reactions, geochemical materials, and forensic medicine [87].

3.3. Spectroscopy-Based Characterization Techniques

Ultraviolet-Visible Region (UV-VIS) Spectrophotometer

Ultraviolet-visible region (UV-VIS) light spectroscopy is a technique used to measure the absorption of absorbed and emitted light and sample in the UV-visible region. UV provides visibility between 200 and 400 nm, while UV-VIS visible region device increases this visibility range up to 400-800 nm thanks to light microscopy. With the development of technology, UV-VIS microscopy provides visibility between 1 nm and 1100 nm wavelength today. The device is a device consisting of a light source, a wavelength selector, detectors, and lenses, respectively. In this device, which provides high resolution, measurement is taken by placing the material being studied between the light source and the detector. The light beam it absorbs is measured before and after the measurement is taken. Accordingly, the lights used in the device are Deuterium and Tungsten lamps. It is a characterization technique used to evaluate the size, concentration, and degree of aggregation of nanoparticles and nanoparticles and to monitor stability. It is a frequently preferred analytical method because it is economical and easy to use. UV-VIS visible light microscopy is also used to determine the number of activated carbon iodine, as well as to prove the accuracy of the amount of material [88]. Since it is sensitive to the near-surface refractive index of nanoparticles and nanoparticles, it is very preferred at the characterization stage. UV-VIS visible zone light microscopy is very useful because it gives easy and accurate results in a short time [89].

• Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is a spectroscopic analysis method using Fourier transform infrared photons used in nanomaterial characterization. FTIR is a characterization technique that examines the bonds between molecules of a material, intramolecular bonds, absorbance or emulsion value [89]. In this technique, it provides information about the functional groups in molecules and the vibrational frequencies of bonds. In this method, which uses infrared rays, the photons sent interact with the conducting material. Fourier transform infrared spectroscopy provides a resolution of 5 microns. It is faster than other methods because the working and measurement time is about 30 minutes. Infrared light is used to determine changes in carbon bonds between carbon-structured nanomaterials and polymers [90]. As mentioned above, the main purpose is to show different absorption and emulsion properties of organic or inorganic materials at different wavelengths using infrared light. While this is being studied in spectroscopy, first of all, nanomaterials in the device, radiation passes through the interferometer and is detected by the detector, creating a signal. This signal is transferred to the computer and the Fourier transform is performed to find samples whose elemental and molecular weight are unknown. Fourier transform infrared spectroscopy is a spectroscopy-based device that is often used in nanomaterial characterization, as it provides a good resolution view in a short time and provides information about molecular bonds.

4. Overview of the Application Areas of Nanobiotechnology

The synthesis, characterization, and material acquisition of nanomaterials bring about a lot of innovations in nanotechnological applications. Today, nanotechnology is considered to be the revolution of the modern scientific era. For this reason, it continues to progress with the studies carried out every day. Nanotechnology is used in most of the items we use in our daily lives, from the clothes we wear, sunglasses, medicines we use, ready-made food packages, technological tools, and hard drives. The reason why nanotechnology is widely used is that it brings advantages such as being cheap, easy to transport, certain content and not taking up much space. In addition, it can be shown as an example that the materials produced can be used repeatedly, the products are quite small, light, and robust [91]. Today, nanobiotechnology is widely used in the food industry, textile industry, automotive industry,

science and education, energy production, and healthcare sector [92], [93].

• Nanobiotechnology in The Healthcare Sector

All studies used nanotechnologically in the field of health are collected under the name nanomedicine. Nanoscale materials and nanoelectronic biosensors have been developed to diagnose, develop treatments, and stop diseases. Currently, the most important approach for the applicability of the correct treatment process in various diseases such as diabetes, cancer, pneumonia, COPD, and Alzheimer's is to make the correct diagnosis [94], [95]. To make an accurate and accurate diagnosis, the importance of nano sensors and nano particles at the nanoscale level is great [96]. For this reason, the main purpose of nano medicine is to support patients whose treatment has not been found with today's technology with nano biotechnology [93]. For this reason, drug release is one of the most common studies in nanomedicine, and a lot of research is being done on this topic. For example, when drugs loaded with nanoparticles are injected into the patient's body, the drug they are carrying is directed toward diseased cells. On the other hand, they pass to healthy cells without harming them and destroy diseased cells. With the acquisition of more information thanks to the delivery of drugs to nanoparticles, the field of nano vaccines has also been developed through many research and development stages. Chemotherapy drugs loaded into nanoparticles are one of the most important application areas [97]. Nano carrier-based application systems enhance cellular and humoral immunity and also have an important value in vaccine treatment [98]. No specially developed treatment has been found for the Covid-19 virus, which has taken the whole world captive in recent years. Instead, nanotechnology and nanomedicine developments have been used to reduce the course of the disease and reduce the mortality rate. It will continue to be an important tool for improving human health and quality of life in future generations as well. The innovations brought by nanotechnology enable the realization of more effective and personalized treatments in the medical world. These structures have many advantages, such as increasing the solubility of drugs, protecting against degradation, reducing toxic effects, extending the duration of activity, improving bioavailability, regulating pharmacokinetic and distribution properties, targeting cells and tissues. The progress of nanotechnology in the field of healthcare brings with it the development of treatments in medicine, while promising an exciting future about the limits of potential future applications. Nanotechnology continues to play an important role in the field of protecting and improving human health [99].

• Nanobiotechnology in The Food Industry

The main goal in the food industry is to increase the number of products that are of high quality, natural, preservative-free, and additive-free, but have a long shelf life for consumers. In line with these requests, nano biotechnology and nanomaterials have also entered the business in the food industry. The primary goal of Nano Food is to make food additives, nutritional supplements, and product packaging. With the use of nanoparticles, products can be produced in packages that are more resistant to environmental conditions and have protective properties against light and gases in the atmosphere. In this way, spoilage-resistant, high-quality, and microbiologically safe foods can be consumed. Another application is that the production, demand and need for recyclable packaging are increasing day by day. These packages are nanomaterial additive products because, in addition to protecting their nutritional content, plastic packaging destroys the carcinogenic effects on nature. Nanotechnological materials in the food industry are very important for environmental sustainability by reducing the waste problem [100]. Thanks to the production of foods using nanotechnology techniques, food processing, and nano-material-based sensors, it contributes to all application areas such as spoilage, loss of consistency, and intelligent packaging.

Nanobiotechnology in Textile Industry

The textile industry is one of the sectors where nanotechnological materials are used the most. With the integration of molecular-scale sensors, electronic devices, and computers into fabrics, fabric production has become possible in the "Interactive Electronic Textile" sector. Fabrics used in the textile industry have been given different properties in the nanometric dimension. As an example of these applications, silver particles have been added to sock yarn to prevent bacteria and bad smell/odors [101]. The production of various types of fabrics that do not wrinkle, do not stain, do not burn, do not tear exists today. In the future, it is aimed to use these fabrics in various fields from medicine to textiles by supporting them with nano materials that change color with sensor mechanisms, are affected by ambient gas, or do not burn. For example, the shirts we wear in everyday life will detect what is happening around us, will be affected by sound, and will record data. Fabrics produced based on nanomaterials are expected to have properties that will store energy, self-clean, and protect against external factors when necessary. The deficiency of smart textile fabrics is that the clothing comfort of the garment is not at the desired level and the need for a power battery is among the problems. If we look at the advantages, the clothing user will be able to receive warnings about dangers and track health information instantly. Nanobiotechnology has a great potential to meet these needs. Promising nano materials are the most useful technological tools to be used in the textile industry in terms of their size, structure, and morphology [102], [103].

Nanobiotechnology in the Automotive Sector

With globalization, competition in the automotive sector is also increasing over time. The interest in vehicles that run on less fuel are durable, long-lasting, use renewable energy sources, and do not harm the environment has increased day by day. The applications offered by nanotechnology show promise in this regard. It has been seen that the motors designed by integrating into nanomaterials have a longer service life and are more powerful. In addition, vehicle parts manufactured with nanomaterials are more robust and lightweight in structure. These nanomaterials introduced to the automotive sector offer as little fuel use as possible and great comfort from a material point of view. In addition, another problem that exists in the automotive sector is that there are very few environmentally friendly, cheap, comfortable-to-use, durable fuel cell alternatives. Fuel cells are systems that convert chemical energy into electrical energy [104], [105]. Nanotechnology supports the production of materials that contribute to the development of fuel cell technology and lead to more efficient sustainable energy production. The advantages that nanotechnology will provide, from hydrogen gas in the automotive sector [106], [107].

• Nanobiotechnology in Science and Education

With the increasing processing speeds and capacity of computers day by day, three-dimensional virtual reality applications have started to be used a lot. With the development of augmented reality technology, the online education environment offers people the opportunity to study as if in a natural classroom environment. This situation creates a cost-effective educational environment that can reduce the financial expenses spent on education, the cost of travel, and food. In addition, with virtual reality, people will be able to Decouple together in the virtual world and increase their social interactions. Another approach is that artists and creative staff will design three-dimensional works of art using virtual reality tools. Engineers and designers have made virtual reality useful for creating product prototypes and conducting simulations. This is why nanotechnology is of great importance in the field of science and education. Because it accelerates innovations, molecular-level investigations, product development, and progress in science and education and offers a better future [108]–[110].

• Nanobiotechnology in Energy Production

Each country aims to obtain nanomaterials that are risk-free, low-cost, and have renewable energy sources. Department of nanobiotechnology, the importance of efficient production, storage, and transportation of energy is quite great [111]. For this reason, the main energy source of nanobiotechnology is provided from hydrogen gas. The reason for this is that hydrogen is very light and flammable. Hydrogen gas has the potential to produce high energy when burned, and as a result, Decontamination of the environment is among the desirable properties. As a result of this, electrical energy is generated when hydrogen gas is burned. Therefore, hydrogen is the candidate that is considered the most suitable for replacing fossil fuels in terms of energy production with its physical structure and chemical reactions [112], [113]. In addition, since hydrogen gas is nanoparticle-based, it is the main energy source that meets the energy conversion, demands and requirements. Studies on energy sources, nano-sized materials, and more efficient, high amounts of energy production has a lot of applications in fuel cells, portable computers, electric vehicles, and mobile phones. With nanobiotechnology, the material size is quite small, safe, cost-effective, easy-to-transport products are being used today and alternative energy sources are being developed [2], [114], [123]–[129], [115]–[122].

5. Conclusion

Nanoparticles, which have gained a different dimension with the technology that has developed since Richard Feynman used the term nano in 1914, are carbon-based materials. Rapidly developing nanomaterials are synthesized using physical and chemical methods. When the material is considered as a whole, it is a top-down approach technique that explains its structural shapes by reducing them to nanometer size using physical methods. Examples of top-down approaches are mechanical milling, electrospinning, lithography, spraying, arc discharge method, and laser ablation method. The method that explains the molecules that exist inside nanomaterials, their dimensional growth, clustering, and synthesis by creating macro molecules, is a bottom-up approach. Chemical vapour deposition (CVD), solvothermal and hydrothermal methods, sol-gel methods, and soft and hard Decoupling are bottom-up approaches that hold and combine by forming macro molecules with the reverse micellar method. After the nanomaterials are synthesized, various analytical techniques are used to detect and measure the nanoparticles. Together with these approaches, the characterization process of materials is carried out based on their optical, morphological, electrical, magnetic, physical, and chemical properties. The characterization of nanomaterials is carried out using microscopic, spectroscopic, and spectrophotometric-based devices. Scanning electron microscopy (SEM), permeable electron microscopy (TEM), and atomic force microscopy (AFM) are used in microscopy-based techniques. In the spectroscopic-based characterization of nanomaterials, various methods such as X-ray crystallography (XRD) are used. In spectrophotometry-based methods, UV-VIS is used for the characterization of nanomaterials in visible light microscopy and Fourier transform infrared spectroscopy (FTIR). Nanomaterials, which have been synthesized and characterized, are currently active in various fields from the textile and food industry, automotive and health sector, science, and education to energy production. From the clothes we wear to the technological tools we use, the nanomaterials that exist in our lives are still being studied by the scientific world and research groups. With these activities, it is aimed to develop existing products or produce new products. With the development of technology, nanomaterials have led to revolutionary changes in our lives.

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