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An Overview of the Historical Development, Synthesis and Characterization Techniques of Nanoparticles Also Used as Radiosensitizers

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ABSTRACT

Today, nanotechnology is widely used in many areas, from medical applications such as targeted drug delivery, cancer treatment, and tissue engineering to energy solutions such as solar panels, fuel cells, and batteries, and environmental applications such as the detection and treatment of air, water, and soil pollution. The use of nanoparticles as radiosensitizer agents in cancer treatment has increased significantly in recent years due to their properties such as high surface area/volume ratio, biocompatibility and targetability. Nanoparticles can enhance the biological effects of ionizing radiation during radiotherapy, thereby aggravating DNA damage in tumor cells and thus increasing treatment efficacy. Metal nanoparticles, especially those with high atomic numbers (e.g., gold, silver, gadolinium), increase photon absorption, increasing local dose density and providing selective radiosensitization at the tumor site. In addition, thanks to surface modifications, targeted delivery, extension of circulation time and protection of healthy tissues are possible. With these aspects, nanoparticle-based radiosensitizers carry significant potential in oncological treatments by contributing to achieving high therapeutic efficacy with lower radiation doses and reducing treatment-related side effects. This review aims to provide a comprehensive overview of the scientific development of nanoparticles used as radiosensitizers, from a historical perspective, and to explain how these structures are classified, synthesized, and characterized. The evolution of nanoparticles is presented in chronological order, starting from prehistoric traces to modern nanotechnology. In the classification section, the grouping of nanoparticles according to their origin, morphology and chemical composition is discussed, while the synthesis approaches are examined on the basis of top-down, bottom-up and hybrid methods. Finally, modern characterization techniques used to determine the structural and surface properties of nanoparticles are included. The compilation aims to provide an up-to-date and systematic resource for researchers working in the field of nanotechnology within the framework of these four main headings.

Keywords: *Nanotechnology, Nanoparticles, Historical Development, Classification, Synthesis Approaches, Characterization Techniques.*

INTRODUCTION

Historical Development of Nanotechnology in the Pre-Modern Age

The earliest historical evidence in nature regarding the combining of atoms and molecules with nanotechnology is thought to have emerged through meteorites after the "Big Bang" that triggered the formation of the universe and the Earth. The most common grain type during the formation of meteorites is nanodiamonds obtained from meteorites [1]. With the discovery and use of fire, the first nanoparticles appeared in the prehistoric period in the form of fullerene, graphene and carbon nanoparticles found in the smoke and soot from fire. Nanoparticles have been produced and used in different historical periods and by various civilizations [2]. For example, cave paintings dating back to 40,000 BC in the Sulawesi caves of Indonesia were created using oil and plant extracts together with carbon nanomaterials [3]. The historical process in which people used nano-level structures without knowing or realizing it, before nanotechnology emerged as a scientific concept, is presented in Figure 1.

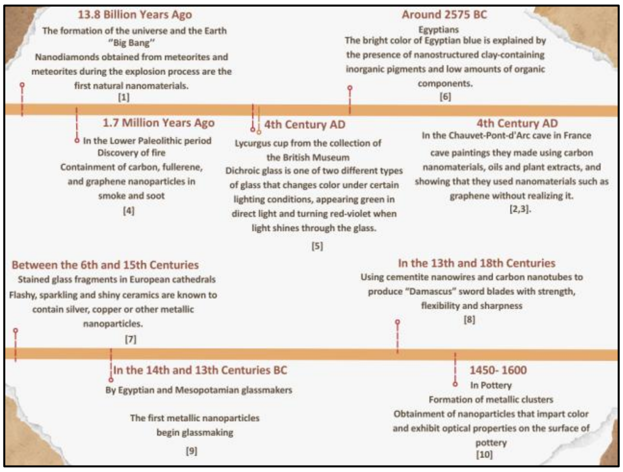

Figure 1: The historical process in which people used nano-level structures without knowing before nanotechnology emerged as a scientific concept.

The development of nanotechnology began with pioneering studies on the behavior of matter at the nanoscale, starting in the mid-19th century. In 1857, Michael Faraday synthesized gold colloidal suspensions called "ruby" and observed that gold nanoparticles caused a color change under certain wavelengths of light, thus demonstrating the first examples of surface plasmon resonance [2]. In the years following these observations, in 1905, Albert Einstein explained Brownian motion and calculated the diameter of the sugar molecule; this study was one of the first quantitative examples of the calculation of molecular scales [2]. In 1908, Gustav Mie theoretically explained the optical properties of light-scattering metallic nanoparticles and made important contributions to the coloration mechanism of colloidal gold particles [11]. In 1920, Irving Langmuir introduced the Langmuir monolayer concept to the literature by defining molecular sequences that can form a single layer on the surface; this approach later formed the basis of surface coating and thin film technologies [12]. In 1925, Richard Zsigmondy was the first to measure the size of nanoparticles such as gold colloids and was one of the first researchers to use the term "nanometer" [12]. In 1928, Edward Synge introduced the idea of optical imaging at nanometric resolution by proposing the system that would later be called the near-field scanning optical microscope (SNOM) [13]. Another major advancement in the field of microscopic imaging occurred in 1931 when Max Knoll and Ernst Ruska developed the transmission electron microscope (TEM) [14]. This development made it possible to observe nanostructures directly. With the invention of the semiconductor transistor by William Shockley, Walter Brattain and John Bardeen in 1947, the microelectronics revolution began and the infrastructure for nanotechnological devices began to form [15]. In the following years, Erwin Müller developed the field-ion microscope in 1951 and became the first scientist to directly image individual atoms on the surface [16]. In 1953, James Watson and Francis Crick revealed the cornerstone of the field of bionanotechnology by analyzing the double-helix structure of DNA [17]. During the same period, in 1956, Arthur von Hippel introduced the concept of molecular engineering, providing the theoretical basis for interdisciplinary nanoscale design [18]. In 1958, Leo Esaki discovered the electron tunneling phenomenon and showed that the behavior of small particles could not be explained by classical physics and that quantum mechanical rules were valid [19].

Historical Development of Nanotechnology in the Modern Age

Richard Feynman made significant and revolutionary contributions to the field of nanotechnology with his talk titled "There's Plenty of Room at the Bottom," which he delivered at the annual meeting of the American Physicists Association on November 29, 1959. Feynman is considered the father of the new field known as "nanotechnology." In his speech, he emphasized that many new discoveries would be possible if production could be made at the atomic and molecular scale, and that unique measurement and production techniques should be developed at the nanoscale. He was awarded the Nobel Prize in Physics in 1965 for predicting the potential of nano-sized particles [20,2]. During the meeting, Feynman presented two problems to the researchers and promised a monetary reward to those who solved them. The first of these was the production of a cube-shaped nanomotor with a side length of 1/64 inch (0.3 mm), and this problem was solved in 1960. The second problem was that the letters had to be shrunk to the point where the entire text of the Encyclopedia Britannica could be written on the head of a nail. Tom Newman, a Stanford University graduate, solved this difficulty in 1985; he succeeded in writing the first page of Charles Dickens's "A Tale of Two Cities" on the head of the nail using an electron beam and received his award [21]. In his speech, Feynman stated that it was interesting that "a physicist or chemist could, in principle, synthesize any chemical substance that he wrote down on a piece of paper," and added: "You can produce that substance by placing the atoms in the places the chemist describes." What he meant here was the existence of machines composed of individual atoms; however, he stated that although the new laws of physics might make the construction of these machines difficult, their re-formation was not impossible [20]. In 1974, Norio Taniguchi first used the term "nanotechnology" (nano-technology) at a conference to describe the stages of creating high-precision semiconductor structures at the nanoscale using atomic layer deposition and targeted ion beam techniques. Taniguchi emphasized that the possibilities of creating new materials and structures by using, combining and manipulating atoms or molecules are unlimited [22,7]. Another important development in the field of nanotechnology was made by Eric Drexler, who earned the first doctorate in molecular nanotechnology at the Massachusetts Institute of Technology. Drexler presented his important vision for the future of nanotechnology in his work titled Engines of Creation: The Coming Era of Nanotechnology, published in 1986 [23]. According to Drexler, "Nanotechnology is the process of controlling the structure of matter at the molecular level, that is, directly manipulating atoms. This technology includes the ability to create molecular systems at the atomic scale and develop nanomachines. He also added, "In fields ranging from medicine to the environment and even space settlement, nanotechnology is the fundamental force that will radically transform our future." These definitions led to a solid foundation of the fundamental principles of molecular engineering and provided important information about the applicability and future potential of nanotechnology in advanced technologies [23, 24]. Table 1 lists the important events that have occurred in the modern era in nanotechnology and the advances made through innovations in many different fields in a chronological order, and these developments are gaining momentum today.

Table 1. Chronological Summary of Important Events Playing a Role in the Development of Nanotechnology and Innovations in Different Fields

History	Event/Development	Reference
1959	Richard Feynman's "There's Plenty of Room at the Bottom" speech	[20]
1974	The term "nanotechnology" was first used by Norio Taniguchi	[20,7]
1981	Gerd Binnig and Heinrich Rohrer of IBM, development of the Scanning Tunneling Microscope (STM)	[25]
1985	Discovery of buckyball (C_{60}–fullerene)	[26,27]
1986	Eric Drexler publishes Engines of Creation Development of Atomic Force Microscopy (AFM)	[23,28]
1991	Discovery of carbon nanotubes by Sumio Iijima	[29]
1996	NASA's initiation of studies in the field of nanotechnology	[30]
1999	Publication of the first nanomedicine book by Robert A. and Jr. Freitas "Nanomedicine"	[31]
2000	Establishment of the National Nanotechnology Initiative (NNI) in the USA	[30]
2004	Andre Geim and Konstantin Novoselov isolate graphene	[32]
2010	Groundbreaking developments in nanomedicine (smart drug delivery systems, cancer treatment)	[33]
2011	Biomimetic synthesis of NPs in the cell membrane	[34]
2018	Nanoscale miniaturization in macromaterials	[35]
2020	NP vaccines (SARS –COV- 2)	[36]
2021	Controllable synthesis of superparamagnetic magnetites in ultrasensitive MRI and angiography	[34]
2022	Genomic editing of CRISPR/Cas9 plasmid DNA in endothelial cells using polymeric NPs	[34]
2023	The proliferation of nanocarriers for cancer cells	[37]

Nanoparticles

Nanoparticles are materials with dimensions typically between 1 and 100 nanometers (1 nm = 10^{-9} m). The nanoscale generally refers to dimensions between 1 and 100 nm [38]. The properties of nanoparticles are determined by the arrangement of atoms and molecules in their structure. During the process of creating, processing, separating and restructuring changes at the atomic or molecular level, nanoparticles may gain new properties while losing some of their properties. These processes increase the structural diversity of nanoparticles and lead to the emergence of a wide range of physicochemical properties such as different shapes, sizes, particle contents, surface interactions, surface charges, mechanical, magnetic, thermal, chemical properties, enzymatic activities, toxicity, biocompatibility, biodegradability and biodistribution. The transition from macro to nano size enables miniaturization of particles. With this reduction in size, the surface area increases and the microscopic features of the objects become more apparent. Many features that cannot be observed at the macro scale can be understood more clearly at the nano scale. Nanoparticles have the ability to mimic the atomic structure in nature, providing the opportunity to examine the world in detail at the atomic or molecular level. They also stand out with their ability to move faster than macro-sized particles. Thanks to these unique properties, nanoparticles are increasingly expanding their use in many sectors, especially in health and medicine, as well as in industry (food, pharmaceuticals, defense), cosmetics, agriculture, textiles, energy and space studies. The usage areas of nanoparticles are summarized in Figure 2.

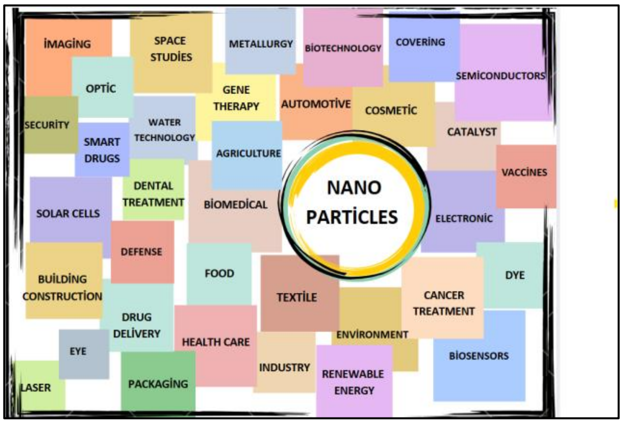

Figure 2: Summary of Uses of Nanoparticles

Classification Of Nanoparticles

One of the most fundamental components of nanotechnology is nanoparticles. In order to understand nanoparticles correctly, they must first be properly classified. These particles, which have various sources including nanoparticles found naturally in nature, those formed accidentally, and those obtained later by artificial methods, are differentiated according to their origin. Additionally, comprehensive classification studies are ongoing based on their size, morphology, structural properties, porosity and chemical composition (Figure 3). [39].

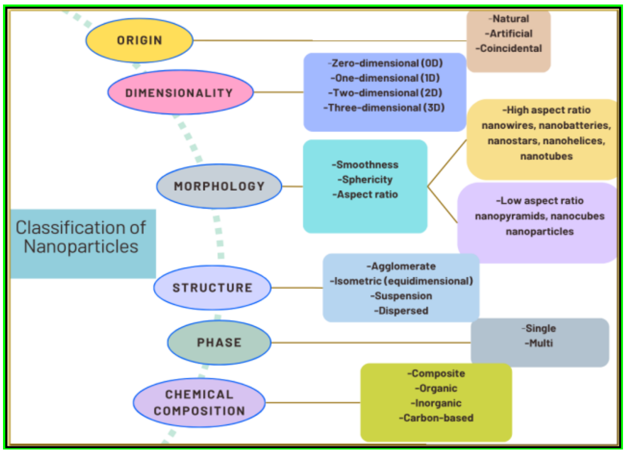

Figure 3: Classification of Nanoparticles

Classification of Nanoparticles Based on Their Origin

Natural Nanoparticles

They are nanoparticles that form as a result of natural processes in nature, without any external intervention. These particles are formed as a result of natural events such as volcanic eruptions or erosion. Examples include tuffs, ashes, combustion products, aerosols (fogs), liquid colloids (blood, milk), vegetal structures (shell, root, flower) and living remains (feather, wing, bone, skin, hair, horn) [2].

Natural nanoparticles such as biogenic iron oxides or silica-based structures derived from diatoms have also been explored for biomedical use, especially as biocompatible scaffolds for targeted radiosensitization and imaging. These naturally occurring nanostructures can be surface-modified to combine low toxicity with high affinity for tumor microenvironments [40, 41].

Artificial Nanoparticles

Artificial nanoparticles are particles with various functions, customized to specific shapes and sizes, produced synthetically under laboratory conditions by imitating the structure of natural nanoparticles. Such nanoparticles are widely used, especially in advanced industrial applications. For example, silica nanoparticles are functionally involved in car tires and calcium carbonate nanoparticles in engine oils [2].

Silica and calcium carbonate nanoparticles exhibit radioadaptive effects, which play a significant role in cancer cell responses to radiation therapy. This significance stems from their ability to adapt to ionizing radiation. Therefore, they have a high potential to render cells resistant or susceptible to radiation therapy [42, 43, 44].

Random Nanoparticles

Nanoparticles are formed directly or indirectly, without an intentional production process, through particles originating from gases, dust, and combustion processes (e.g., exhaust emissions, sudden braking) [2, 45].

Classification of Nanoparticles Based on Their Size

One of the most fundamental features that distinguish nanoparticles is their size. Many physical and chemical properties are directly related to particle size. Particle size is an important factor that determines dynamic behaviors such as diffusion coefficient and speed, sedimentation rate and amount, and electrical mobility. Therefore, size control greatly affects the application areas and functionality of nanoparticles [46].

The size distribution of nanomaterials affects their surface area, making them attractive for use in biological contexts.This increases their high mobility within the body, facilitating their transport to organs and penetration into relevant tissues.It enables effective targeting.In medical imaging studies, nanoparticles are known to enhance tumor diagnosis and surgical procedures by enhancing image contrast when delivered to specific tissues [47].

The use of nanoparticles in radiotherapy offers benefits by enhancing ionizing effects. A study investigated the radiosensitization effects of needle-shaped nanocarrier titanate nanotubes (TiONts) on human glioblastoma cell lines (SNB-19 and U87MG) at low and wide dose ranges. The results indicated that TiONts exhibit radiosensitizing effects at low and high doses, inhibit DNA damage repair, and cause cell arrest in the G2/M phase. The size and shape characteristics of nanoparticles suggest they may be effective in various cancer types [48].

Zero (0) Dimensional Nanoparticles

Nanoparticles in this group are defined as structures whose dimensions (height, width, depth) are all on the nanometer scale, that is, all three dimensions are smaller than 100 nm. It is one of the most common types of nanoparticles. Examples include metal nanoparticles, inorganic quantum dots, fullerenes, magnetic nanoparticles, and polymer-based nanoparticles [49].

Metal nanoparticles, inorganic quantum dots, fullerenes, magnetic nanoparticles, and polymer-based nanoparticles exhibit radioluminescence for various applications in radiation detection, nuclear security, and biomedical imaging. The stability, optical, resonance, and contrast properties of nanoparticles offer potential for radiation detection and improved efficiencies [50, 51].

One (1) Dimensional Nanoparticles

One-dimensional nanostructures are structural elements with two dimensions at the nanoscale (1–100 nm) and the third dimension at the macroscopic scale. These structures are generally in the form of nanowires, nanotubes, nanopillars and nanofibers, and their lengths are much greater than the other two dimensions. Their electrical, thermal and mechanical properties vary with size and they are widely used in nanoelectronics, energy transmission, sensors and composite materials [49].

Nanotubes, nanoparticles, and nanofibers hold promise for enhancing radiosensitizing effects in cancer treatments. For example, nanotubes increase radiation absorption, making cancer cells more susceptible to potential damage in treatment strategies. Nanofibers are used as radiosensitizers by increasing the dose of radiosensitizing agents due to the high energy density and energy absorption coefficients of high atomic number nanomaterials. They are also a key structure in drug delivery mechanisms. Nanofiber-based biosensors are being designed and used to monitor the outcomes of radioactive stimulation treatments. Nanostructures, with their targeted delivery and radiation absorption, offer promising approaches to radiosensitization studies [52, 53, 54].

Two (2) Dimensional Nanoparticles

They are particles with only one dimension at the nanoscale and the other two dimensions are not at the nanoscale. Although their thickness is on the nanometer scale, their surface dimensions generally exceed 100 nm. Graphene and graphene oxide, boron-based nanolayers, nanofilms and nanocoatings are evaluated in this class [49].

Graphene and graphene oxide nanostructures are an important factor in assessing the radioactive effects and stimulatory effects of radiofrequency exposure, enabling the appropriate interaction between radioactive and radioactive irradiation. They offer innovative therapeutic strategies and biodiagnostic benefits [55, 56].

Three (3) Dimensional Nanoparticles

Although all component dimensions of these structures are larger than 100 nm, they are particles that exhibit properties at the nanoscale by forming three-dimensional volumetric structures. They are non-porous or low-porosity structures. Nanofiber bundles, nanostructured graphene derivatives and nanocomposite materials are included in this group [57].

Nanofiber bundles, nanostructured graphene derivatives and nanocomposite nanoparticles, especially nanocomposites, with their intrinsic radioprotective properties, are creating exciting developments in protecting healthy tissues from damage caused by unavoidable radiation during radiotherapy by obtaining nanoradioprotective agents [58].

Classification of Nanoparticles Based on Their Morphology

Nanoparticles can consist of a single component or a combination of multiple components; therefore, their morphological properties—that is, shape, surface roughness, and geometric structure—are of great importance to their functionality. The roughness of the particles, their spatial position (e.g. aspect ratio), different size and shape variations directly affect these morphological properties. The most fundamental and determining mechanism in the formation of the morphology of nanoparticles is the nucleation process. This process begins with molecules, atoms or ions forming a certain order in the reaction environment within the liquid phase, creating nanostructure nuclei. In the first stage of nucleation, rapid and reversible formation of crystal structures occurs at a certain energy level; in the second stage, as the system exceeds the critical energy level, an irreversible phase change occurs and the nucleus grows and acquires a stable structure. This process is shaped by the type of nucleation (homogeneous or heterogeneous), ambient temperature, degree of saturation, crystallization rate, reaction volume, ionic strength of the medium, surface absorption capacity, nuclear growth rate, free energy change of this rate and the presence of surfactant [59].

High atomic number (Z) nanomaterials are preferred as radiosensitizers due to their X-ray attenuation coefficients, which increase their radiotherapeutic efficacy. Because they play a significant role in ROS generation, their presence is associated with short lifespan and localized exposure. Furthermore, the radiosensitizing effects of nanomaterials depend on their size, morphology, and surface chemistry. Properties such as strong covalent bonds between molecules, weak van der Wall interactions between layers, and narrow band gaps enable nanomaterials to rapidly transport charge. Therefore, radiosensitization has positive consequences on the cytotoxicity, targeting capabilities, and biodistribution of effective therapies [60].

Classification of Nanoparticles According to Their Structure

The structural properties of nanoparticles are of great importance, especially in terms of their interaction behavior in agglomerate (clustered) and aggregate (tightly bonded powder structure) forms. Aggregates have higher surface energy compared to agglomerates and exhibit more active behavior within the system by performing interparticle bonding mechanisms more quickly and effectively [61]. Nanoparticles are also divided into three groups according to their porosity levels: micro, meso and macroporous structures. Micropores have narrow pores with diameters less than 2 nm and interact highly with small molecules due to their low diffusion kinetics. Mesopores have diameters ranging from 2–50 nm and provide a suitable interaction surface for medium-sized molecules. Macropores have diameters over 50 nm and are capable of interacting with large structures such as biological macromolecules; for example, nanogels and nanotubes are considered in this group [62]. In addition, the crystallinity properties of nanoparticles are also important in structural classification: monocrystalline structures consist of an orderly and uniform arrangement of atoms, polycrystalline structures contain orderly but differently oriented regions, and amorphous structures do not have a specific crystal lattice pattern. In addition, colloidal properties such as zeta potential, which determines the dispersion stability of nanoparticles in liquid media, are also critical for biological and physicochemical effects [63].

Micro-, meso-, and macroporous nanostructures offer numerous advantages due to their versatility in surface modifications for drug delivery. The porous channels of porous materials have substantially low solubility. Therefore, they offer solutions to significant challenges in drug capture and delivery approaches and treatment methods. Their pore diameters and large surface areas facilitate key steps such as absorption, dosage cycles, and the number of applications. In studies, combined interactions have been created, positively altering radiosensitive effects [64].

Classification of Nanoparticles According to Their Phases

Nanoparticles are generally classified as single-phase or multi-phase systems in terms of their crystalline phase structure. Metal nanoparticles, graphene oxide and carbon nanotubes are considered as single-phase nanoparticles because they mostly have a single-layered and homogeneous structure. These structures are preferred especially in electronics, optoelectronics and sensor technologies due to their properties such as high conductivity and structural stability. On the other hand, multilayered core-shell type nanoparticles are classified as dual-phase with their structures consisting of two different materials. While the core of these structures usually carries a functional property (e.g. magnetic property), the outer shell is designed to provide additional functions such as biocompatibility, stability or targetability. These multiphase structures provide great advantages, especially in areas such as drug delivery systems, targeted therapy (e.g., selective delivery to cancer cells), imaging agents, and catalysis applications. This diversity in phase structures has a direct impact on the chemical reactivity of nanoparticles, their surface modification potential, optical properties and interactions with biological systems. In particular, phase separation in the core-shell structure enables the development of multifunctional nanocarrier systems by allowing different functions to be combined on a single platform [65].

Nanoprobes and sensors are being designed to assess changes in biomarkers in cancer cells resulting from the use of relevant antibodies in cancer stem cells responsible for cancer regeneration in various cancer types, including breast, brain, lung, colon, and skin cancer. This design increases cell capture and release efficiency and also enables single-cell molecular profiling [66].

Classification of Nanoparticles Based on Their Chemical Composition

Composite Nanoparticles

They are complex nanoparticles that occur in different shapes and compositions. They are a type of surface-modified nanocomposites obtained by combining an organic shell and an inorganic core composed of metal or metal oxide. They can be organic, metal, or carbon-based [67].

In radiotherapy, metal organic frameworks (MOFs), organic linkers, and metal ions (especially those with high atomic numbers) are gaining attention for increasing radiosensitivity. Radiosensitizing agents are being developed. They overcome radioresistance and improve therapeutic outcomes [68].

Organic Nanoparticles

Organic nanoparticles are colloidal structures consisting of carbohydrates, lipids and various natural or synthetic organic compounds, generally ranging in size from 10 nm to 1000 nm. Dendrimers, micelles, ferritin structures and liposomes are examples of this group. These structures generally show high compatibility with biological systems. Due to their high water solubility, changeable surface morphology and targeted transport capacity, they are widely used in biomedical applications, especially in cancer treatment, gene delivery and vaccine development. In addition, their low toxicity, their lack of immunogenic effects and their biodegradability in the body make organic nanoparticles indispensable in the field of biotechnology [69].

Dendrimers, micelles, ferritin scaffolds, and liposomes serve as effective radiosensitizers, increasing the sensitivity of cancer cells to radiation therapy. Micelles, self-assembling nanostructures, utilize liposomes to release their cargo in response to radiation, resulting in increased cancer cell sensitivity. Ferritin-based radiosensitizers utilize the accumulation of naturally occurring ferritin to effectively deliver therapeutic agents to cancer cells [70, 71, 72].

Inorganic Nanoparticles

Inorganic nanoparticles are structures that do not contain organic structures and are generally composed of metal, metal oxide, ceramic and semiconductor components. Their hydrophilic properties, high chemical stability and generally low toxicity make these particles advantageous in environmental and industrial applications. It is widely used in areas such as imaging agents (MRI contrast agents), sensors, biosensors, energy storage systems and photocatalysis. Components such as gold, silver and silica are among the most frequently used inorganic structure examples [73].

Nanoparticles such as gold, silver, and silica are frequently used as radiosensitizers due to their unique photoelectric decay properties. They provide high dose capacity and high therapeutic effects. Radiosensitization facilitates active targeting by nuclear-localized cellular uptake and localization within or near the nucleus [74].

Metal Nanoparticles

Metal nanoparticles represent nanoscale forms of pure metals. Structurally, they can be produced in different geometric forms such as sphere, rod, cylinder, quadrilateral or hexagonal. These particles, which can be synthesized by reduction-oxidation (redox) reactions, have unique properties such as electrical conductivity, surface plasmon resonance and high surface energy. Therefore, they are used in many fields such as electronics, photonics, medical imaging, antimicrobial surface coatings and catalytic reactions. The most commonly used metal nanoparticles include gold (Au), silver (Ag), iron (Fe), zinc (Zn), copper (Cu), aluminum (Al) and cadmium (Cd) [73].

ROS are present in all cells and are essential for cell signaling and function. They are one of the dominant mechanisms in radiation therapy using low linear energy transfer (LET), such as X-rays. Metal nanoparticle radiosensitizers such as gold, silver, iron, zinc, copper, aluminum, and cadmium interact physically with ionizing radiation. These processes result in the photoelectric effect, Compton scattering, Auger electron emission, X-ray fluorescence, and pair production. These are the mechanisms that generate reactive species, including ROS. This provides insight into the physicochemical basis of radiosensitization and radioprotection [75].

Metal Oxide Nanoparticles

The high reactivity of metal nanoparticles causes them to oxidize over time and turn into metal oxide nanoparticles. The properties of these structures can vary depending on the pure metal form. Examples include iron oxide (Fe_2O_3), silicon dioxide (SiO_2), zinc oxide (ZnO), titanium oxide (TiO_2), and aluminum oxide (Al_2O_3). Since these nanoparticles have advantages such as photocatalytic activity, UV blocking, antimicrobial properties and biocompatibility, they are used in a wide variety of areas such as sunscreens, water purification systems, environmental sensors, biosensors and drug delivery platforms [73].

The mechanism underlying the radiosensitizing effects of Fe_2O_3, SiO_2, ZnO, TiO_2, and Al_2O_3 nanoparticles is that the dense core of the nanoparticles produces secondary electrons that increase the local absorbed dose, and the organic shell of the nanoparticle causes damage within the cell, leading to increased ROS formation when the core-catalyzed oxidative stress occurs. This changes the type of irradiation [76].

Semiconductor Nanoparticles

Semiconductor nanoparticles have a transition structure between metallic and nonmetallic properties and have diameters in the range of 1–20 nm. These particles stand out especially in their forms known as quantum dots. Since their electrical conductivity varies in response to external stimuli such as temperature, light and electric field, they are used in many technological applications such as photovoltaic cells, LED technologies, photodetectors, biosensors and imaging systems. Compounds such as cadmium selenide (CdSe), zinc sulfide (ZnS) and gallium arsenide (GaAs) are examples of this group [69].

CdSe, ZnS and GaAs nanoparticles are being used to determine the uptake and accumulation amounts of reactive oxygen species, and applications are being developed to provide better results in biomedical imaging, targeted drug delivery and radiosensitizer effects [77, 78].

Ceramic Nanoparticles

Ceramic nanoparticles are defined as non-metallic solids and generally contain inorganic structures composed of oxides of metals or metalloids such as titanium, calcium and aluminum. These nanoparticles are generally found in polycrystalline, porous, amorphous and hollow structures. Thanks to their properties such as high mechanical strength, chemical inertness, high temperature resistance and electrical insulation, they are widely used in a wide variety of fields such as biomedical implants, tissue engineering scaffolds, energy storage systems (e.g. batteries) and environmental filtration systems. In addition, their high loading capacity offers remarkable advantages in drug delivery systems [79].

The radiosensitizing effects of nanoparticles composed of metal oxides or metalloids such as titanium, calcium, and aluminum indicate that nanoparticles can be used as imaging and therapeutic agents, increasing the dose delivery efficiency and creating new implications for radiotherapy treatments [80].

The ionizing radiosensitization effects of zinc-doped hydroxyapatite (HA) nanoparticles synthesized by microwave-assisted irradiation were investigated on breast cancer cells. While HA was observed to reduce crystal size and theoretical densities, the addition of Zn2+ ions to the HA structure increased irradiation efficiency [81].

Radiosensitivity changes were evaluated in human glioblastoma (U251) breast tumor and brain metastatic tumor (MDA-MB-231BR) cells using hydroxyapatite nanoparticles (nano-HAPs) synthesized by the sol-coagel method with a size of ~50 nm. As a result, it was reported that they caused significant radiosensitization [82].

Carbon-Based Nanoparticles

Carbon-based nanoparticles are special structures composed solely of carbon atoms and are of central importance in nanotechnology due to their extraordinary physical and chemical properties. Included in this class are graphene, fullerenes (C_{60}, C_{70}), carbon black nanoparticles, and carbon nanotubes (CNTs). These structures are generally known for their high biocompatibility, low toxicity, high stability, excellent mechanical strength and hydrophilic surface properties [83]. Fullerenes (C_{60} and C_{70}) are hollow, single or multilayered, spherical structures, usually 7–7.6 nm in diameter, formed by pentagonal and hexagonal carbon rings. It is used in areas such as electron transfer, drug delivery and photodynamic therapy. Carbon black nanoparticles are hard, spherical structures consisting of amorphous carbon atoms with a diameter of 20–70 nm. They are used as industrial additives as pigments, fillers and electrical conductors. CNTs are cylindrical tubular structures with a diameter of 1–2 nm. They are divided into three different types according to the structure they form by folding onto themselves: Single-walled nanotubes: These are the smallest in diameter, usually ~0.7 nm in diameter, and are monolayered. Double-walled nanotubes: Formed by the interweaving of two carbon layers. Multi-walled nanotubes: They are multi-layered structures with diameters up to 100 nm and attract attention with their high mechanical strength and conductivity properties [62]. Carbon-based nanoparticles are widely used in areas such as neurological targeting, gene delivery, drug delivery, biosensors, conductive polymers and energy storage technologies [69].

Single-walled carbon nanotubes (hollow, with high specific surface areas) are being created. Carbon nanotubes, which have the potential to penetrate biological barriers with high efficiency, control nanoparticles accumulation and retention in the tumor tissue. Therefore, they are known to have high radiosensitization effects [84].

1. SYNTHESIS OF NANOPARTICLES

The synthesis method and production conditions of nanoparticles are largely determined by the target size range and the physical and chemical properties of the particles. It is necessary to create the desired nanoscale structures, determine the specific properties of these structures and select appropriate reaction mechanisms regardless of their sources. In addition, the controlled execution of these mechanisms and their stopping when necessary are possible through the application of effective synthesis methods. The main purpose of the synthesis approaches is to narrow the size range of the nanoparticles and achieve close and homogeneous distributions. Figure 4 shows the synthesis approaches of nanoparticles.

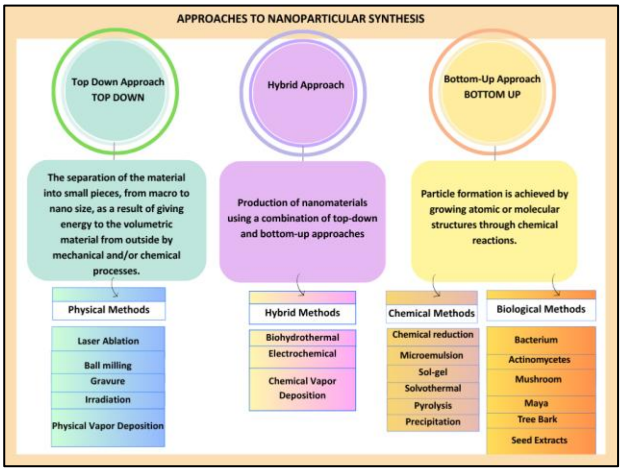

Figure 4: Approaches Used in the Synthesis of Nanoparticles

Top Down Approach

The top-down synthesis approach refers to the disintegration of macroscopic structures into small atomic fragments or nanoparticles by externally applied energy sources (such as mechanical, chemical, thermal or radiation). This method is not suitable for materials with soft texture and is relatively costly. However, it is widely preferred in the production of nanoparticles of various sizes, complex geometries and morphological properties. The most commonly used techniques include nanolithography, laser ablation, thermal dissociation, mechanical etching-milling and sputtering methods (Figure 4) [85, 86].

Nanolithography

Nanolithography is a technique used to write and print nanoscale patterns on photosensitive materials. Patterning is of great importance in fundamental cellular research. It is also used in the creation of functional machine parts at the microscopic level through microelectromechanical systems. Nanolithography relies on the self-organization and agglomeration properties of molecular building blocks to create designs that enhance their performance. Application areas are divided into different techniques such as photolithography, X-ray lithography, nanoimprint lithography, light-coupling nanolithography, electron beam lithography, extreme ultraviolet lithography, Dip-Pen nanolithography and scanning probe microscopy lithography according to their resolution, efficiency and cost [87].

Laser Ablation (Photoablation)

Laser ablation is the process of removing material from solid or liquid surfaces in a controlled manner using laser beam. The effectiveness of this method varies depending on the wavelength of the laser used, the depth at which the energy is absorbed, the application time and the amount of material. Laser ablation technology is also widely used in surgical applications, artificial intelligence-assisted cutting processes, and machine learning applications [88].

Thermal Decomposition (Thermolysis)

Thermal decomposition or thermolysis is a decomposition method in which the structure and contents of a material are changed by breaking the physical integrity and chemical bonds between its components under the influence of heat. With this technique, the degradation degree and chemical composition of the material are changed in a controlled manner, thus obtaining the desired nanoparticle properties [89].

Mechanical Erosion and Milling

Mechanical abrasion-milling is a physical disintegration method used to bring the material to a certain fineness. It is especially preferred in the production of composite, metallic and ceramic nanoparticles. In this technique, free-moving balls continuously break up the material and turn it into powder. Process efficiency and quality of results depend on parameters such as particle size, type of energy used (low or high), milling speed, ambient conditions, processing time, temperature, added substances and composition [90].

Spraying Method

Spraying is a coating technology widely used especially in the pharmaceutical and food industries. With this method, material coatings of different shapes are achieved by accumulating a suspension of nanoparticles. The process consists of four stages: preparation of the suspension, creation of atomic order, drying, and detachment of the material onto the surface. As atomic-scale droplets collect on the surface, the coating is completed by evaporation of the solvent. The quality and thickness of the coating are controlled by factors such as the viscosity of the liquid used, temperature, spray speed and low solvent waste level [91,92].

Hybrid Approach

The hybrid approach has been based on the observation that particles formed in natural erosion processes (e.g. volcanic erosion) are generally initially structured irregularly and randomly. It is essential to use both top-down and bottom-up methods together to create controlled and adjustable nano-sized structures from these particles. Thus, by combining the advantages of both methods, nanoparticles with higher quality and specific properties are obtained. The most commonly used techniques in this context include biohydrothermal method, electrochemical synthesis and chemical vapor deposition (CVD) (Figure 4) [2].

Bio-Hydrothermal Method

Biohydrothermal synthesis is a process carried out in an aqueous solution environment in a closed reaction vessel. In this method, the reaction environment is created by increasing the pressure and temperature in a controlled manner and the molecules are allowed to dissolve and crystallize in the solution. In the crystal growth process, the bonds between molecules, the bond energy, the geometric configuration of the solvent and solute, the temperature and pressure of the environment and the surface structure play a decisive role. These parameters directly affect the morphology and size of the crystals formed [93].

Chemical Vapor Deposition

Chemical vapor deposition method involves chemical reaction of a volatile precursor on the substrate and its surface to form thin films or nanoparticle layers. This technique plays an important role, especially in biomedical applications, due to its biocompatibility and its capacity to prevent the accumulation of biological material. With this method, homogeneous, controlled and functional coatings can be obtained on surfaces [94].

Bottom-Up (Creation and Construction) Approach

The bottom-up approach is the process of creating nano-sized particles by the controlled combination of atoms or molecules through chemical or physical interactions. In this method, starting materials can be in solid, liquid or gas phase. This technique, which is frequently applied to naturally occurring materials, enables the obtaining of nanoparticles with narrow size distribution, homogeneous composition and safer in terms of toxicity. The most commonly used bottom-up techniques include sol-gel method, microemulsion, pyrolysis and biosynthesis (Figure 4) [95, 96, 97, 73].

Sol-Gel Method

The sol-gel method is a comprehensive synthesis technique that generally consists of two steps. In the first step, colloidal-sized solid particles (sol) are formed using precursors such as metal nitrates or salts. In the second stage, these colloidal particles combine and form a gel. This method occurs at low temperatures and provides high productivity, superior surface finish, chemical reactivity and material quality. The sol-gel method is an important technique frequently used in nanomaterial production, whose effectiveness increases with the decrease in impurity rates of the materials [98, 99].

Microemulsion Method

The microemulsion method allows effective control of the droplet size, composition and shape of nanoparticles. This method is carried out on isotropic systems consisting of immiscible liquid phases, by means of surfactants (e.g. water, oil). In addition to surfactants, co-surfactants are also used, which form flexible and mobile layers. Microemulsions have dispersed domains with a diameter of 10-50 nm, and these domains are generally interconnected or spherical in structure. These systems function as nanoreactors, providing shape and size control of nanoparticles in a thermodynamically stable environment [100].

Pyrolysis (Thermal Decomposition)

Pyrolysis is a thermodynamic technique used to produce nanoparticles through the controlled chemical degradation of organic materials by exposing them to high temperatures in an inert atmosphere. In this method, parameters such as temperature, pressure, gas phase conditions, application time, humidity and raw material amount are decisive on the process quality and efficiency. Pyrolysis can be applied in different types, including slow, medium and fast, and is an effective method for converting organic materials into nanostructured form [101].

Biosynthesis (Green Synthesis)

Biosynthesis is the production of nanoparticles by utilizing the natural metabolic processes of living organisms (such as plants, fungi, algae, bacteria) or their inactive forms. This method is an environmentally friendly, repeatable and cost-effective process that does not require special and complex conditions. For biosynthesis, firstly the biological material to be used is washed appropriately, dried and boiled with pure water to obtain the extract. This extract is stored at low temperature and mixed with metal salts to form nanoparticles with the help of a magnetic stirrer. Biosynthesis has become an important method in recent years due to its low risk of contamination and suitability for large-scale production [102, 103,104].

Characterization Techniques Of Nanoparticles

Precise characterization of the size distribution, shape, surface chemistry, crystal structure and other physicochemical properties of nanoparticles is of great importance for material design, performance and long-term application safety in the field of nanotechnology. A complete knowledge of the chemical, physical and biological properties of nanoparticles enables the determination of different application areas depending on the techniques and methods used. In this way, reliable modeling can be done in scientific research and economically efficient strategies can be developed. Furthermore, accurate assessment of the potential toxicity and ecotoxicity effects of nanoparticles enables accurate risk analyses in terms of human health and environmental safety. In summary, the more comprehensive and detailed the characterization of the sample, the greater the accuracy and applicability of scientific interpretations [105]. A comprehensive summary of nanoparticle characterization techniques is presented in Figure 5.

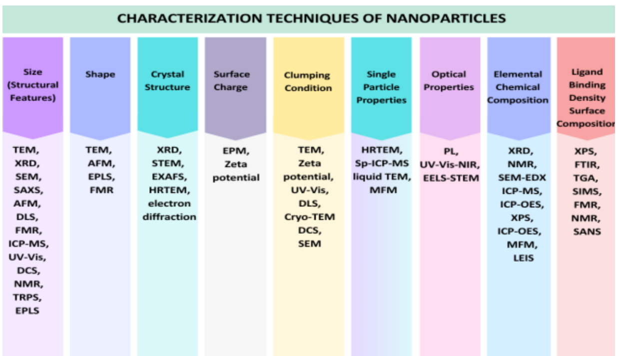

Figure 5: Commonly Used Techniques in the Characterization of Nanoparticles. (Transmission

Electron Microscopy (TEM), X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Small-angle X-ray scattering (SAXS), Atomic Force Microscopy (AFM), Dynamic Light Scattering (DLS), ferromagnetic resonance technique (FMR), Inductively coupled plasma-mass spectrometry (ICP-MS), Ultraviolet-visible spectroscopy (UV-vis), Differential Centrifugal Sedimentation (DCS), Nuclear magnetic resonance (NMR), Tunable resistive pulse sensing (TRPS), Elliptically polarized light scattering (EPLS), scanning transmission electron microscopy (STEM), extended X-ray absorption fine structure (EXAFS), cryogenic electron transmission electron microscopy (cryo-TEM), High-resolution transmission electron microscopy (HRTEM), single particle-inductively coupled plasma-mass spectrometry (SP-ICP-MS), Magnetic force microscopy (MFM), photoluminescence spectroscopy (PL), Energy dispersive X-ray spectroscopy (EDX), X-ray photoelectron spectroscopy (XPS), Fourier Transform Infrared Spectroscopy (FTIR), thermogravimetric analysis (TGA), ion mass spectrometry (SIMS), small angle neutron scattering (SANS)).

Transmission Electron Microscope

Transmission Electron Microscopy (TEM) is a high-resolution imaging technique widely used in the analysis of interactions at the molecular level in tissue and cell compositions. In TEM, electrons produced under vacuum by the electron gun at the top of the microscope are focused into a very thin beam with the help of electromagnetic lenses and passed through very thin sections of the sample. As electrons pass through the sample, they either scatter or strike the fluorescent screen directly, creating images of various hues depending on different density and composition. This technique provides detailed, three-dimensional visualization of the internal structure of the sample. It is effective in examining the interior of the sample rather than the surface and provides valuable information in analyses such as biodistribution and particle uptake [106].

X-Ray Diffraction

X-ray diffraction (XRD) is an analytical technique based on the principle of constructive interference resulting from the interaction of monochromatic X-rays with samples having a crystalline structure. X-rays are produced by the cathode ray tube and made monochromatic by appropriate filters and directed to the sample. Constructive interference occurs when X-rays diffracted by atoms in the crystal structure of the sample satisfy the conditions of Bragg's law ($n\lambda = 2d \sin\theta$). This is related to the interatomic distance (d-spacing) and diffraction angle in the sample. Analyzing the diffraction pattern allows the crystal structure and mineral species of the material to be determined. The XRD technique provides comprehensive information by reaching different crystal orientations when the sample is in powder form. The obtained diffraction patterns are analyzed by comparing with standard reference databases [107].

Scanning Electron Microscope

Scanning Electron Microscope (SEM) is a surface analysis device that works by focusing electrons accelerated under high voltage onto the sample surface. As the electron beam is scanned over the sample surface, various interactions occur between the electrons and the sample atoms. The signals resulting from these interactions are collected by detectors, passed through signal amplifiers, converted into an image signal and presented as a high-resolution image on the monitor. SEM reveals the morphological structure, topography and composition of the sample surface in detail. The signals received by the sensors are converted into digital form and analyzed and visualized in a computer environment [108].

Field Emission Scanning Electron Microscope

Field Emission Scanning Electron Microscopy (FE-SEM) is an imaging technique similar to SEM but with an improved electron source and beam generation method. In FE-SEM, the sample surface is scanned with a thin electron beam focused by electromagnetic lenses; in this process, electrons reflected or interacting with the surface are detected, creating high-resolution images of the sample's surface topography and morphology. The most important difference from SEM is the electron production system. FE-SEMs use a Field Emission Gun (FEG) as the electron source. FEG produces a very thin and intense electron beam by applying a high electric potential gradient. In this way, FE-SEM provides the advantage of higher resolution, lower energy analysis and revealing more detailed surface features compared to traditional SEM. Thanks to these features, FE-SEM plays a critical role in surface analysis in nanotechnology, materials science and biomedical fields [109].

Atomic Force Microscope

Atomic Force Microscopy (AFM) is a sensitive microscopy method used to examine the morphological and mechanical properties of the sample surface at the nanometer scale. AFM uses a mechanical tip to measure the mechanical profile of the surface, its roughness and topography at the atomic and molecular level. This tip is mounted on a piezoelectric material composed of piezoelectric crystals and capable of three-dimensional translation. By applying electrical polarization, the piezo material changes shape with nanometer precision and moves the lever. In this way, the mechanical tip scans the sample surface and creates a topographic map of the surface. AFM can also measure the electric potential at the surface of the sample when conductive levers are used, thus providing detailed information about the electrical properties of the surface. With its high resolution and direct surface measurement capability, AFM is widely preferred in the characterization of nanomaterials, the study of biomolecules and the analysis of surface interactions [110].

Dynamic Light Scattering

Dynamic Light Scattering (DLS) is an effective physical characterization method used especially in determining the size distribution of nanomaterials in liquid media. This technique is based on measuring the fluctuations in the intensity and time course of laser light scattered from small particles in dilute solutions. As particles undergo Brownian motion, the intensity of the scattered light varies with time. By analyzing these density changes, the diffusion coefficients of the particles are calculated and the hydrodynamic diameters of the particles are determined through the Stokes-Einstein equation. DLS provides rapid and reliable information on the average size, size distribution and agglomeration state of nanoparticles. Additionally, parameters such as viscosity and temperature of the measurement medium also play an important role in this analysis [111].

Scanning Near Optical Microscope

Scanning Near Optical Microscopy (SNOM) is an advanced microscopy technique that can perform imaging at the nanometer scale, exceeding the limit of optical resolution. While the diffraction limit, which determines the resolution limit of conventional optical microscopes, is constrained by the wavelength of light, SNOM overcomes this limit by using near-field optical interactions. The technique is based on a very thin, pointed tip covered in metal. This tip is much smaller in diameter than the wavelength of light and is held very close to the sample surface (about a few nanometers). The proximity of the tip is precisely adjusted by feedback piezoelectric control systems. Light emitted from the tip of the tip (Light emitted from the probe tip) interacts with the electric field on the sample surface, providing high-resolution optical information about the sample. With this method, it is possible to study nanoscale optical properties, fluorescence, and local electric field distribution. SNOM finds a wide range of applications, from materials science to biology, from nanotechnology to semiconductor technology [112].

Zeta Potential

Zeta potential is a quantitative indicator of the “effective” electric charge on the surface of nanoparticles in a colloidal system and is one of the key determinants of colloidal stability. This parameter represents the intensity of the electrostatic repulsive forces between particles; high absolute zeta potential values (positive or negative) indicate that the repulsion between particles is strong, thus ensuring that the system remains stable without agglomeration. The measurement is usually carried out in a special cell containing electrodes. After the solution is added to this cell, the charged nanoparticles move towards the electrode with the opposite charge, thanks to the voltage applied to the cell. The speed of the particles during this motion is reflected in the frequency fluctuations observed in the intensity of the scattered light as the laser beam is passed through the solution. The speed of movement of particles at different applied voltage values is measured and the zeta potential is calculated using this data. Zeta potential measurements play a critical role in the analysis of surface chemistry, dispersion properties, biocompatibility and toxicology of nanoparticles [111].

Fourier Transform Infrared Spectroscopy

Fourier transform infrared spectroscopy (FTIR) is based on the principle that covalent bonds in molecules selectively absorb infrared (IR) radiation at specific wavelengths. This IR radiation absorbed by covalent bonds changes the vibrational energy of the bonds within the molecule, and these vibrations can be in the form of stretching or bending. The type of bond and the chemical structure of the atoms within it determine the frequency of these vibrations. Therefore, each molecule and functional group has a unique IR absorption spectrum. FTIR is widely used in the determination of molecular structure, chemical composition and surface functional groups. It provides detailed analysis of the surface chemistry of cellular molecules and nanomaterials, especially in biomedical applications [113].

Raman Spectroscopy

Raman spectroscopy is based on the inelastic scattering of light (Raman scattering) incident on a sample. In this process, as photons are scattered from the sample, the energy of some photons changes by interacting with the molecular vibrations and rotational motions of the sample, so that the frequencies of the scattered photons differ from the original photons. This frequency change creates a spectrum that is like a fingerprint specific to the molecular structure. Raman spectroscopy is used to study the chemical bonds and structures of molecules and is complementary to IR spectroscopy. It is preferred for the characterization of nanomaterials, especially carbon-based structures (e.g., graphene, nanotubes) and crystal structure analysis [114].

CONCLUSION

Nanoparticles, as the basic building blocks of the field of nanotechnology, are of critical importance in many interdisciplinary fields such as materials science, biomedical applications, energy storage and environmental technologies, thanks to their unique physicochemical and morphological properties [115, 116]. In this review, information about the historical development of nanoparticles is given, and the systematic classification of nanoparticles according to their origin, size, morphological and structural properties, synthesis methods and advanced characterization techniques are discussed. Detailed characterization of the structural, morphological, chemical and physicochemical properties of nanoparticles is of great importance for the sustainable development of the field of nanotechnology. A full understanding of these properties is of critical importance for effectively determining the usage areas of nanoparticles, minimizing toxicity and ecotoxicity risks, and performing technical and economic modeling [105]. The diversity of nanoparticle synthesis methods, both top-down and bottom-up approaches, as well as the development of hybrid techniques, enable the controlled optimization of material properties. Particularly, achieving precise control over morphology and phase properties increases the application potential [86, 2]. In recent years, environmental sustainability and biocompatibility criteria have become increasingly important in the production of nanomaterials. In this context, biological synthesis (green synthesis) methods offer ecologically sensitive and economical production processes by minimizing the use of toxic chemicals and the risk of environmental pollution [102, 103]. Green synthesis, as a reproducible and scalable approach using plant extracts, microorganisms and other biological agents, attracts attention with its advantages of high biocompatibility and functionalization in biomedical applications [117]. In this respect, green synthesis, as a pioneer of the environmental approach in nanotechnology, has the potential to create new opportunities in both health and the environment. In order to develop sustainable and safe applications in the field of nanotechnology in the future, the structural and chemical properties of nanoparticles must be systematically classified, synthesis protocols must be precisely standardized, and multi-scale characterization techniques must be disseminated. In addition, comprehensive investigation of the biophysical behavior and toxicological effects of these structures will contribute to the design and application of nanotechnological products in compliance with ethical, environmental and social responsibilities.

Radiosensitizers consist of compounds and nano-sized structures that have a significant effect on the destruction of cancer cells through the effects of ionizing radiation. They serve as therapeutic agents in radiation-based treatments. New nanoparticle-supported approaches are being developed for the diagnosis and treatment of complex cancers. The goal of these approaches is to increase the sensitivity of cancer cells to radiation-induced destruction and reduce the potential for harmful effects on healthy cells during this process. Therefore, combinations of nano-radiosensitizers and radiosensitizing NPs hold promise in various cancer treatments[118].

Recent studies have shown that integrating RT effects with nanoparticles (NPs) in conjunction with imaging systems is creating intriguing possibilities in the field of precision oncology. Therefore, targeting or targeting cancer cells represents a highly innovative concept. Nanoparticles with high atomic number (Z) elements, such as gold, are being used to increase radiation sensitivity due to their unique physicochemical properties, such as increased radiation energy absorption, known to induce intense cell damage, and their ease of manipulation. This results in increased tumor selectivity in treatments using RT and NP combined, and the tumoricidal effect is significantly enhanced compared to RT alone [119].

Conflict of Interest

There are no conflicts of interest and no acknowledgements.

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