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Article

Investigation of Ferromagnetic Nanoparticles' Behavior in a Radio Frequency Electromagnetic Field for Medical Applications

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Abstract: This work raises the hypothesis that it is possible to use ferromagnetic carbon nanotubes filled with iron to hyperthermally destroy cancer cells in a radiofrequency electromagnetic field. This paper describes the synthesis process of iron-filled multi-walled carbon nanotubes (Fe-MWCNTs) and presents a study of their magnetic properties. Fe-MWCNTs were synthesized by catalytic chemical vapor deposition (CCVD). Appropriate functionalization properties of the nanoparticles for biomedical applications were used, and their magnetic properties were studied to determine the heat generation efficiency induced by exposure of the particles to an external electromagnetic field. The response of the samples was measured for 45 min of exposure. The results showed an increase in sample temperature that was proportional to concentration. The results of laboratory work were compared to the simulation using COMSOL software.

Keywords: multi-walled carbon nanotubes filled with iron; catalytic chemical vapor deposition; COMSOL Multiphysics simulations; ferromagnetic materials

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1. Introduction

Research on modern cancer treatment methods is a crucial area of scientific investigation worldwide. Despite significant advancements in the field of medicine, challenges still persist in cancer detection and treatment. Poland faces one of the highest cancer mortality rates among OECD (The Organisation for Economic Co-operation and Development) countries, with breast, prostate, lung, and colorectal cancers being the leading causes of cancer-related deaths [1]. According to the International Agency for Research on Cancer, in 2018, 17.0 million new cases of cancer and 9.5 million people worldwide died from cancer, and they predict that by 2040, the number of new cases will rise to 27.5 million worldwide, while deaths will rise to 16.3 million [2]. These statistics seem quite frightening, as the number of cases is trending upward so far. The main problem with cancer is detection; in many cases, the patient learns of the disease when it is too late for non-invasive treatment or even for fully effective treatment, not even mentioning potential metastasis. The second problem is that cancer can attack basically any system and any organ.

Continuous efforts in scientific development and interdisciplinary collaboration between various fields of science, including automation, electronics, electrical engineering, space technologies, medicine, and materials engineering, have led to the development of innovative cancer treatment methods. One breakthrough achievement in this regard is the use of carbon nanotubes (CNTs). These nanomaterial structures have an extensive surface area suitable for attaching ferromagnetic nanoparticles [3–5].

Since the discovery of carbon nanotubes in 1991, nanocarbon nanoparticles have garnered significant attention in various technological fields [6]. Carbon nanotubes are a type of carbon nano-allotrope composed of sp2 hybridized carbon atoms arranged in a hexagonal pattern, forming hollow tubular structures [7]. These nanotubes can be single-walled, double-walled, or multi-walled. Single-walled carbon nanotubes typically have a diameter of about 1 nm and can be up to 100 nm in length, giving them distinctive dimensions, morphology, and chirality [8,9]. Their optical, electronic, mechanical, and physical properties have been extensively studied. These unique structures possess high surface areas and notable structural and physical characteristics [6,10]. Carbon nanotubes are synthesized using various methods, including chemical vapor deposition, laser ablation, arc discharge, catalytic growth, and organic routes. They have found applications in numerous practical sectors such as energy devices, sensors, electronics, membranes, engineering structures, and the aerospace, automotive, and biomedical fields [7,9,10].

Carbon nanotubes find a number of medical applications. After proper functionalization (surface modification), they become completely biocompatible. CNTs can cross biological barriers as new drug delivery systems. Carbon nanotubes containing drugs, antibodies, proteins, or DNA molecules can serve as carriers that transport the drug directly to diseased tissues. At first, the possibility of using such carriers in cancer therapy and in the treatment of viral infections was primarily investigated. Now, the possibility of using CNTs as carriers in immunotherapy or gene therapy has also been demonstrated [11].

Magnetic Particle Hyperthermia (MPH) is a form of cancer treatment based on the use of heat induced by magnetic nanoparticles (magnetic nanoparticles—MNPs) [12]. Considering that cancer cells are more sensitive than healthy cells in the temperature range of 41–47 °C, increasing the temperature within this range is believed to inhibit the growth of cancer cells and cause tumor shrinkage [13]. Hyperthermia therapy aims to destroy the tumor by triggering apoptosis [14,15] (i.e., programmed cell death). Necrosis also occurs due to disruptions in microcirculation and other mechanisms, depending on the treatment duration and temperature. Ferromagnetic nanoparticles have enormous potential in cancer treatment. Their ability to harness magnetic fields for targeted drug delivery, magnetic hyperthermia, and immunomodulation represents a groundbreaking shift in the approach to cancer treatment. Magnetic nanoparticles coated with special ligands (special proteins detectable by cancerous cells' receptors) are able to selectively link to cells requiring treatment. Ongoing advancements in nanotechnology, coupled with meticulous research, are likely to pave the way for new and effective treatment methods that leverage the capabilities of ferromagnetic nanoparticles to enhance patient outcomes [16].

A very promising and extremely important application of carbon nanotubes that is directly related to the topic of this article could be the use of them to selectively destroy cells (especially cancer cells). For this purpose, CNTs filled with ferromagnetic material (e.g., iron) are delivered, similar to drug delivery systems, to diseased cells [17]. The nanotubes are then heated externally by thermal ablation, which causes an increase in the cell's temperature and its death from excess heat [18,19]. In this way, only diseased cells are destroyed without invasively affecting the rest of the organism [17–21]. Exposing cells to temperatures higher than 42◦C causes cell death, because above this temperature proteins coagulate [22,23].

The aim of this work was to investigate the possibility of using carbon nanomaterials in medicine, characterizing their properties and functionalizing them for application in thermal ablation therapy, as well as observing the phenomena occurring in ferromagnetic nanocontainers placed in a radio frequency electromagnetic field. An analysis on the possibility of heat generation in elements of magnetic nanoparticles shows that it would be possible to use them in cancer therapy for the treatment of malignant tumors located anywhere in the body, provided that the nanocarriers (ferromagnetic carbon nanotubes) that will change RF energy into heat are attached exclusively to the cancer cells.

This article is a continuation of the research described in the paper "Examination of the Effect of RF Field on Fe-MWCNTs and Their Application in Medicine" [24].

2. Materials and Methods

2.1. Synthesis of Fe-MWCNTs

The process of synthesis of iron-filled multi-walled carbon nanotubes (Fe-MWCNTs) by catalytic chemical vapor deposition (CCVD) is described in papers [24–29].

Carbon nanotubes were synthesized by catalytic chemical vapor deposition (CCVD). The apparatus used in the synthesis process was a three-zone horizontal furnace, allowing temperature control in each zone. The furnace consisted of three temperature zones to provide thermal insulation. Zone I (573 K) was the catalyst solution evaporation zone, Zone II was 573 K, and Zone III was the deposition zone in the temperature range 1020–1120 K. The carrier gas for the process was argon. A quartz tube was placed inside the furnace, inside which (in the deposition zone) a silicon substrate covered with a nanometer layer of SiO_2 was placed. To create a neutral atmosphere (initial conditions), the flow of two gases was switched on: argon and helium. The system also required a hydrogen flow for the synthesis process. The system was equipped with a precision substrate dosing system—a Medima S2 pump (from Medima Sp. z o.o., Warsaw, Poland). A solution of ferrocene with xylene (concentration 200 g/L) was used as the reaction substrate. Ferrocene ($Fe(C_5H_5)_2$) was the source of carbon and iron was the reaction catalyst, while xylene (C_8H_{10}) was the carbon source. The mixture was injected externally at an inlet velocity of 16 mL/h (Figure 1).

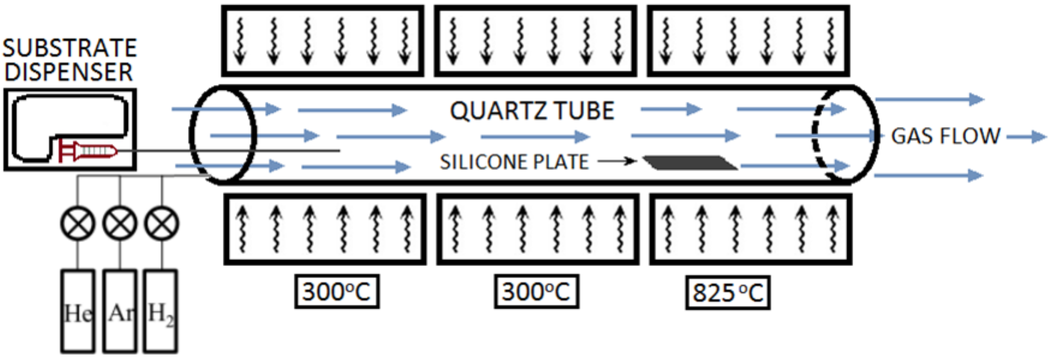

Figure 1. Schematic of the system used for the synthesis of carbon nanotubes by the CCVD method [25].

During previous research, a system for the electrothermal method of production carbon nanotubes filled with ferromagnetic material was developed. This enabled the production of Ferromagnetic Nanocarriers using the catalytic chemical vapor deposition (CCVD) method. The resulting nanoparticles were multi-walled carbon nanotubes filled with iron, with a diameter of 40 nm, a length of 100 µm, an iron content of 20.44%, and a purity of 63%.

The process was assumed to consist of four stages:

1. Purification of the quartz reactor from oxidizing material.
2. Establishment of initial conditions (temperature, gas flow) at a selected level.
3. The actual synthesis process of Fe-MWCNTs.
4. Cooling.

The content was analyzed using thermogravimetric analysis (TGA), followed by an examination of the remaining material with SEM-EDS (Figure 2). TGA results indicated that the fabricated carbon nanotubes (CNTs) were predominantly multi-walled. The material was also analyzed using XRD, which showed that the iron content in the CNTs consisted of both α-Fe and γ-Fe phases. This was evidenced by the diffraction peaks corresponding to the 111 reflections of γ-Fe and the 110 reflections of α-Fe. The iron content was determined to be 59% γ-Fe and 41% α-Fe. No Fe_3C was detected in the samples.

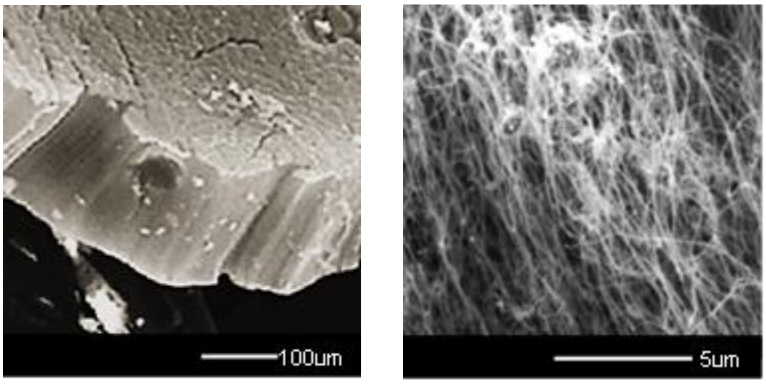

Figure 2. SEM images of fabricated Fe-MWCNTs (left—magnification ×250; right—magnification ×10,000).

2.2. Investigation the Heating Quality of Fe-MWCNTs

The next stage of the work was to investigate the heating quality of the obtained material. To investigate the heating capabilities of Fe-MWCNTs, the temperature rise in magnetic liquids was measured after exposure to electromagnetic fields of 20 kA/m at five frequencies (110, 165, 330, 428, 650 kHz, and 1 MHz). This study was designed to compare the results using different concentrations in different suspensions. A description of the experiment as well as the experimental set up is included in the following papers: [24,27,30].

The results of the laboratory experiments were then compared to the simulation of a heating phenomenon using COMSOL Multiphysics 5.0 software.

3. Results

To investigate the heating capabilities of Fe-MWCNTs, studies on the temperature increase in magnetic liquids were conducted after exposing them to an electromagnetic field of various frequencies selected from the RF frequency range.

In ferromagnetic elements of extremely small sizes (such as Fe-MWCNTs), there is a possibility to generate energy based on hysteresis loops, viscous dissipation, and Brownian and Neel relaxation losses. Considering that these phenomena pertain to individual elements and the structure of this work adopted a simplification that considered a solution mimicking the human body (human tissues) with suspended Fe-MWCNTs as the object, it was determined that in such an approach, eddy currents would most effectively influence energy generation. This approach introduced an error to the results, but due to the ratio of the volume of the human body to Fe-MWCNTs in magnetic hyperthermal heating, it was considered negligible.

The main goal of this stage of the work was to observe the behavior of CNTs under the influence of a radio frequency electromagnetic field and to find the best conditions for the highest heating rate. All measurements had the most similar conditions possible each time. The samples were observed for 45 min. The experiments were conducted in a solution thermally isolated from the environment by placing the test sample in an electromagnetic field at frequencies of 110 kHz, 165 kHz, 330 kHz, 428 kHz, 650 kHz, and 1 MHz.

In Figure 3, all measurement results are collectively gathered for comparative purposes. This chart includes information on the maximum values for verification. The dashed line represents the protein coagulation temperature. As can be seen, the higher the iron content, the greater the temperature increase.

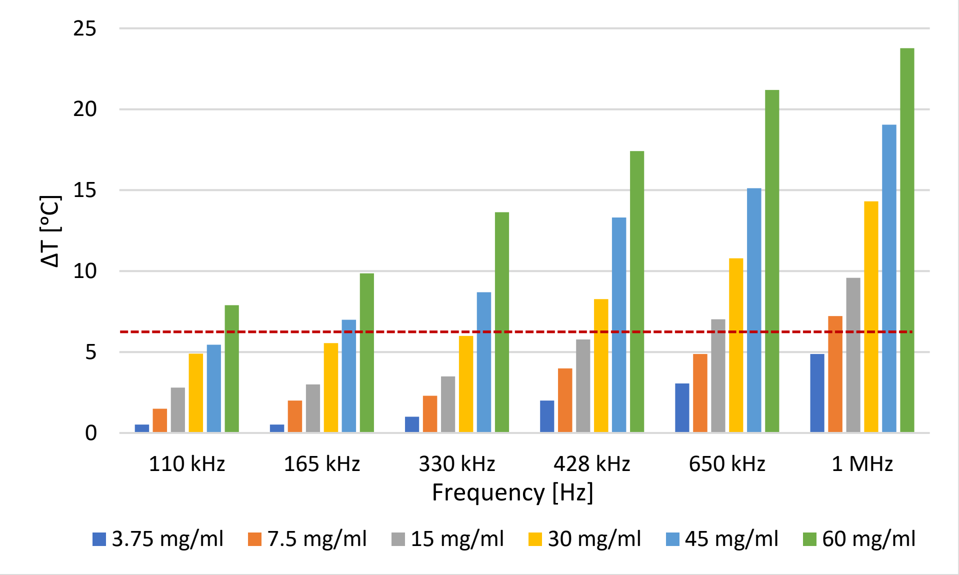

Figure 3. Bar graph of all measurements.

The highest temperature difference among all of the conducted studies was observed in sample number 36, which experienced a temperature increase of nearly 24 degrees. This sample had a concentration of 60 mg of Fe-MWCNTs per 1 mL of water in an electromagnetic field of 1 MHz frequency. The conditions of this measurement confirmed the initial and most intuitive assumption. It was assumed that since iron nanoparticles inside Fe-MWCNTs are materials that heat up, a higher concentration would also increase the temperature change. Exactly as hypothesized, a higher concentration of Fe-MWCNTs in the samples resulted in a greater temperature change. Across all six electromagnetic field frequencies, the best results were obtained for the highest concentrations. However, these concentrations were entirely dependent on the amount of components in each sample. Even though logically increasing the concentration would provide better results, dispersion was the main problem. Two attempts were made to prepare samples with higher concentrations. Unfortunately, they were so dense that properly mixing the components to achieve a uniform distribution was very problematic. For this reason, measurements carried out on samples with higher concentrations were not sufficiently reliable and were not considered. The general behavior pattern was that the higher the concentration, the greater the increase in temperature.

The second variable was the frequency of the electromagnetic field acting on the sample. The logical assumption was that a higher frequency would cause the sample to heat up faster, which also proved to be true. All tested frequencies yielded satisfactory results to some extent. A higher frequency always had the greatest heating speed. Interestingly, in the case of the two lower frequencies, the results were not always as expected. First, the difference between temperature changes was much smaller than when comparing two higher frequencies, but this was the result of a frequency difference of only 58 kHz, whereas, for the second pair, the frequency was almost twice as high.

The research findings demonstrated that as the concentration of CNTs increased, along with an increase in frequency, the heating rate also increased. Consequently, it was established that this method holds promise for therapy using the produced CNTs since the sample temperature exceeded 43 °C, which is considered sufficient for cancer cell necrosis. In Figure 3, all measurement results are summarized for comparison. For verification purposes, this graph includes information on the maximum values. The highest temperature difference in all the tests carried out was obtained in the study of sample No. 36, whose temperature increased by almost 24 degrees. This was a sample with a concentration of 60 mg of Fe-MWCNTs per 1 mL of water in an electromagnetic field with a frequency of 1 MHz.

Due to the direction of activities aimed at faithfully reflecting reality, the model included a blood vessel. Models were created in the COMSOL environment (Figure 4), which can be simplistically described as an external system of coils generating a magnetic field with nanocontainers placed inside the coils in a tissue environment or so-called experimental environment (reflecting nanocontainers suspended, for example, in water). It is important to note that, in pursuit of a faithful reflection of reality, coils were used instead of an external magnetic field, implemented as boundary conditions. The models had to account for the coils being cooled with water at a specified temperature and flow rate.

In hyperthermia treatment, the electromagnetic field in the tissue is generated by applicators (antennas—a system of coils, ablative electrodes) surrounding or adjacent to the tissues. The number and configuration of these applicators are among the research tasks that need to be developed appropriately for each specific therapeutic goal. The role of the coil system is to transform the voltage of pulses from the electrical pulse generator into a local magnetic field in the tissues. It must be remembered that the efficiency of the process depends on the balance between the rate of thermal energy formation and the dissipation of this heat in the tissue.

Mathematical models for thermal problems in hyperthermia are too complicated to be easily solved. Most of the unresolved problems in the medical field are described by nonlinear partial differential equations.

This work utilized the Heat Transfer Module, which allows for the simulation of heat transfer associated with heat convection, heat conduction, and radiative heat exchange. Additionally, the program is equipped with modules considering the impact of heat related to metabolism and blood perfusion. This provides the opportunity to examine heat flow and the effect of current flow on temperature distribution in living tissue.

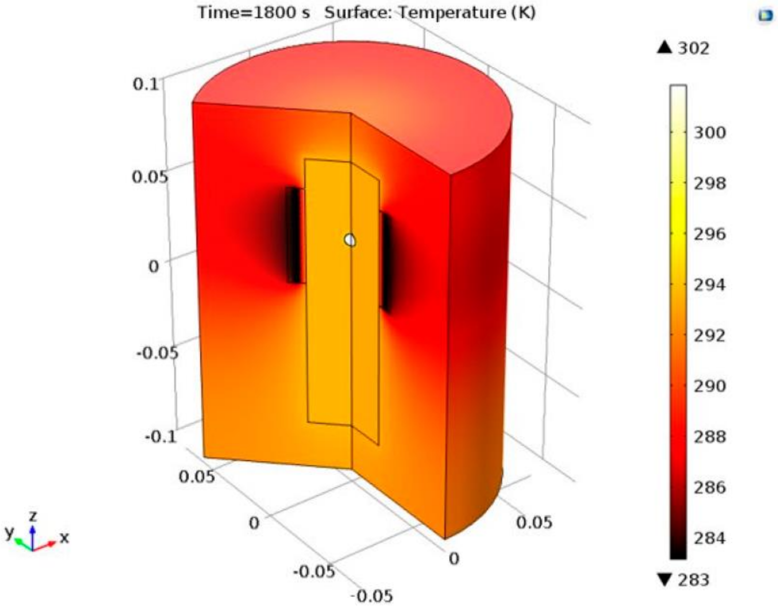

Figure 4. Computer simulation of heat distribution in the sample.

Figure 5 presents a comparison of all temperature increase results obtained in simulations depending on the intensity and frequency of the field after 40 min. The possibility of heat generation was examined at a magnetic field strength ranging from 10 kA/m to 60 kA/m with frequencies ranging from 110 kHz to 1 MHz. Figure 6 shows a bar chart illustrating how much time is needed to reach the targeted temperature.

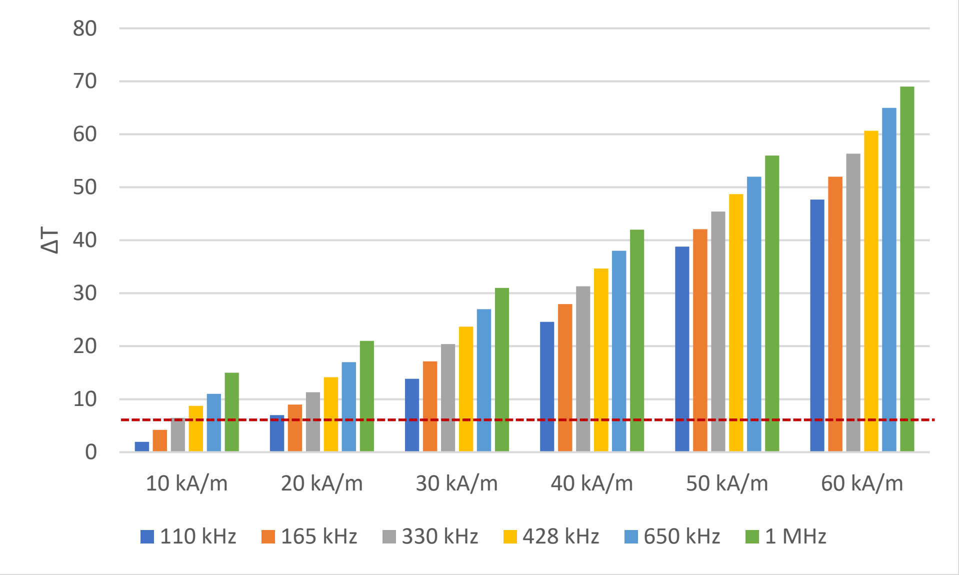

Figure 5. Summary of all temperature rise results depending on the intensity and frequency of the field after 45 min (the red line shows the temperature of protein coagulation).

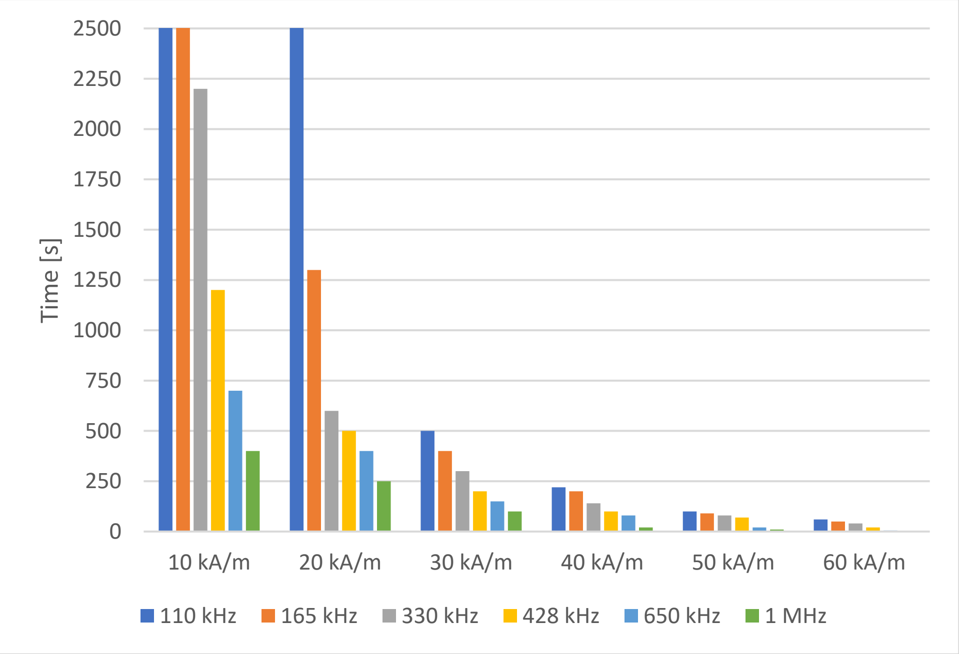

Figure 6. The time it takes to reach a temperature increase of 7 degrees—crossing the limit of protein coagulation (values for the first two samples for 10 kA/m and for the first sample for 20 kA/m did not reach this value).

Based on the results obtained, it was found that there is a clear correlation between field strength and temperature rise, with higher field strength at higher frequencies leading to greater temperature rise. In addition, the time required to reach the maximum temperature also depends on the field strength—the higher the field strength, the faster the sample heats up, and the limit of reaching the maximum temperature value and reaching the plateau occurs later.

4. Discussion

Modelling the phenomenon of CNT heating suspended in a liquid simulating the human body showed that increasing the intensity of the electromagnetic field significantly enhances the therapy's efficiency (increases the temperature gradient). A maximum temperature increase of 69 °C was achieved. However, achieving such a concentrated suspension is challenging in practice. Additionally, there is uncertainty regarding how many of the suspended CNTs will attach to cancer cells and participate in hyperthermal therapy. Estimating these parameters will be a significant milestone; it is potentially the subject of future research. Nevertheless, the results obtained under laboratory conditions with a field intensity of up to 20 kA/m and a frequency of 110 kHz were already sufficient. Hence, they received the greatest attention during subsequent work.

Theoretical considerations were made regarding the potential application of CNTs and the method for conducting therapy on living organisms. This research demonstrated that frequencies in the range of 100 kHz and 1 MHz effectively induced cancer cell necrosis. However, the final determination of the optimal power of the CNT production device will only be possible through "in vivo" experiments on animals, which should be pursued as a separate research project. In this phase of research, it will be necessary to estimate the number of nanocarriers used in therapy to effectively destroy cells and determine the precise parameters of the field used in the proposed method. The successful completion of this phase is crucial and paves the way for in vivo studies. At this stage, the quantity and concentration of nanoparticles suspended in a biocompatible solution must be determined, and exposure parameters must be well defined to focus on the cellular response to the heating agent.

5. Conclusions

This investigation confirmed the principle regarding the hyperthermia phenomenon and showed that the created simulation model is an effective tool for predicting and analyzing this phenomenon. The results obtained from the simulation were consistent with those obtained in laboratory conditions, but the use of the simulation model made it possible to study the phenomenon in a more detailed and extended manner. It was observed that changing the field strength significantly affected the heating rate, which allowed the sample to be heated to the required temperature above the limits of protein coagulation (43.6 °C) in as little as a few seconds, which solves the problem of prolonged exposures to electromagnetic fields and keeps this time within the limits set by the FDA (Food and Drug Administration) for SAR (Specific Absorption Rate).

The results obtained from this experiment provide valuable information that provides a good reference point for further research. One of the goals is to develop an effective dispersion method to further study the heating properties of the suspension at a given concentration and to begin testing cell lines. In this phase, it will be necessary to estimate the number of nanocarriers used in the therapy to effectively destroy cells and to determine the exact field parameters used in the proposed method. Successful completion of this phase is crucial and paves the way for in vitro studies. In this phase, the amount and concentration of nanoparticles suspended in a biocompatible solution should be determined and the exposure parameters should be well defined to focus on the cellular response to the heating medium.

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