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Biosynthesis and Study of Microwave Radiation Absorption by Ilmenite Nanoparticles

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ABSTRACT

Nanostructured radar absorbing materials (RAMs) have received steadily growing interest because of their fascinating properties and various applications compared with the bulk or micronized counterparts. These nanostructured materials have increased surface area, number of dangling bond atoms and unsaturated co-ordination on surface lead to interface polarization, multiple scatter and absorbing more microwave. In this paper, new method was developed to produce ilmenite nanoparticles. Ilmenite nanoparticles were synthesized in the laboratory under microbial environment in presence of low magnetic field. This process could provide an easy and inexpensive method for the preparation of ilmenite nanoparticles. They possess remarkable electrical, mechanical, optical, thermal and chemical properties and low cost material which make them a perfect fit for many engineering applications. Biologically synthesized ilmenite nanoparticles were used as microwave absorbent material to study the microwave properties like reflection coefficient and return loss. The results showed that these nanostructured ilmenite particles can be used to design multilayer radar absorbing materials.
Keywords:- Biosynthesis, ilmenite nanoparticles , microbes , microwave absorbance, RAMs.

I. INTRODUCTION

Nanocrystals (NCs) or nanoparticle (NPs) have different physical and chemical properties from their bulk materials[1-3]. They also have high surface to volume ratio which differs the characteristics of the nanocrystals or nanoparticles from the bulk materials[3-5]. The unique properties of nanocrystals or nanoparticles can be utilized in the applications like optoelectronics, energy conversion, magnetic storage or nanomedicine [6-12]. Synthesis of NCs or NPs with different structures, compositions and sizes is achieved for many type of compounds like chalcogenides, transition metal oxides, lanthanide base compounds, actinides based compounds and noble metals [13-17].

Radio absorbing materials (RAMs) have properties of absorbing microwave energy and reducing electromagnetic backscatter. They can be used in many applications like stealth technology of aircrafts, television image interference of high-rise buildings and microwave dark room and protection [18-19]. RAMs are designed to suppress the reflected electromagnetic energy incident on the surface of the absorber by dissipating the magnetic or electric fields of the wave into heat. Excellent RAMs exhibit properties like strong microwave absorption with a high magnetic and electric loss [20-22]. Nanostructured RAMs also exhibit strong microwave absorption. There are many types of nanostructured RAMs like nanocrystal RAMs, core-shell nanocomposite RAMs, nanocomposite of MWCNT and inorganic materials RAMs, nanocomposite of nanostructured carbon and polymer RAMs. These properties of nanoparticles (NPs) lead to interface polarization and multiple scatter, which is useful to absorb more microwave.

In this paper, ilmenite NPs can be formed by using nanobiotechnology techniques. In nanobiotechnology, inorganic structures can be synthesized under mild pH, pressure and temperature. There are examples of microorganisms which synthesize inorganic materials include magnetotactic bacteria (magnetite nanoparticles),diatoms (siliceous materials) and S-layer bacteria (gypsum and calcium carbonate layers).

Here we have used a new technique for the preparation of ilmenite NPs. It is a biological technique in which microbes (bacteria) were concentrated around ilmenite mineral under magnetic field to synthesize ilmenite NPs of particles size ranging from 10nm to 40nm. The crystallography and particle sizes of all the fabricated compounds are confirmed by XRD and small angle X-ray scattering (SAXS) methods.

II. SYNTHESIS OF NANOSTRUCTURES BY MICROORGANISMS LIKE BACTERIA

Microorganisms such as bacteria, fungi, yeast, actinomycete, algae and virus all can be used to synthesize nanomaterials which include metal, semiconductor, quantum dots and alloy with different sizes and shapes.

Microorganisms like bacteria were utilized to synthesize nanoparticles earlier. Due to the mild conditions, high yield and easy purification, bacteria become the most widely studied microorganism for the synthesis of nanomaterials.

Bacteria like *B. subtilis* 168, *B. licheniformis*, *E. coli* DH5α, *Pseudomonas aeruginosa* and *S. algae* are used to synthesize Au nanoparticles. The synthesis of silver nanoparticles are also reported with *B. licheniformis* and *B. thuringiensis*. Nanoparticles of metal oxides like cobalt oxide nanostructures were synthesized with the Gram-positive bacteria, *B. subtilis*. Synthesis of Titanium dioxide nanoparticles are also reported using the microbes like *B. subtilis* and *Lactobacillus sp*.(Gram-positive bacterium).

Researchers also utilized *E. coli*, *S. algae* to synthesize nanoparticles of compounds cadmium sulfide, cadmium telluride, platinum nanoparticles.

Aerobic *Actinobacter sp*. (Gram-negative bacteria) used to synthesize nanoparticles iron oxide nanoparticles, silicon/silica nanocomposites.

Magnetospirillum gryphiswaldense is first to be discovered that could synthesize intracellular magnetic nanoparticles.

Geobacter sulfurreducens is used to synthesize metal nanoparticles of Pd(II).

The synthesis of silver and selenium nanoparticles have been reported using *Morganella psychrotolerans and Klebsiella pneumonia*. There are also many other bacteria that could be utilized to synthesize different nanoparticles. UO2 nanoparticles were formed by *Shewanella oneidensis* MR-1 depending on the c-type cytochromes.

So, microbes like bacteria can be used to synthesize the nanoparticles of compounds because these microbes can control the pH, temperature, chemical potential and other required parameters for required chemical process for formation of nanoparticles [23].

III. OBJECTIVES

1) Biosynthesis of ilmenite NPs.
2) To study the microwave absorption of the synthesized ilmenite NPs.

IV. METHODOLOGY

Ferrites like $FeTiO_3$ are considered to be the best magnetic material for electromagnetic wave absorbers due to their excellent magnetic and dielectric properties. Ilmenite is antiferromagnetic (AFM) and insulting material and composites of ilmenite can be a semiconductor. It is an abundant material in Earth crust . It can be used in many applications like inspintronics, chemical catalyst, absorbing material, optoelectronics etc. due to its wide band gap and absorbing properties. It is the raw material for producing TiO2 and Ti and low-cost, promising oxygen carrier for solid fuels combustion in a chemical-looping combustion (CLC) system. Ilmenite nanoparticles can be synthesized by sol-gel method, mechanochemical method, ball milling method etc [24].

In our research, ilmenite nanoparticles were synthesized in the laboratory. To synthesize the required material, 100gm of ilmenite ($FeTiO_3$) is kept in a cylindrical container made up of non magnetic material. The atmospheric O_2 plays an important role in this experiment. Due to atmospheric O_2, it has been seen that there are phase transitions in $FeTiO_3$.

All the following microbes such as Stenotrophomonas maltophilia, Pseudomonas putida, Pseudomonas aeroginosa, Enterobacter cloacae, Staphylococcus sciuri, Acinectobacter cacloaceticus, Pantoeau agglomerans, and Flavobacterium spp. are together concentrated around $FeTiO_3$ in that container. The experimental container which contains $FeTiO_3$ and microbes is kept under low magnetic field of around 10^{-2} tesla. Under magnetic field, it was observed in some research paper that microbes move along a specific direction and they show some unique characteristics [25,26]. The experiment is carried out for 12 hours at room temperature 27◦C. After 12 hours the sample was taken out from the experimental container. Then XRD and SAXS is carried out on the prepared sample which confirms the formation of Ilmenite ($FeTiO_3$) NPs. The microbial system concentrated around $FeTiO_3$ played an important role to combine the molecules to form $FeTiO_3$ NPs. These microbes control the temperature, pH value, chemical potential of the reaction and various other parameters like physical environment to carry out chemical reaction [27-28]. The systematic arrangement of the experiment is shown in the Fig. 1.

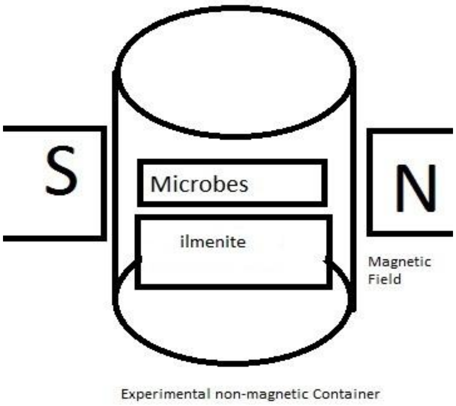

Fig. 1 Experimental arrangement for synthesis of ilmenite nanoparticles

V. INTERPRETATION OF PARTICLE SIZE BY XRD ANALYSIS

The ilmenite nanoparticles were studied using XRD (XRD-panalytical, operated at 40 kV and 30 mA, Cu-K∝). A series of intensive peaks (2θ/deg: 32.64, 35.33,40.43,48.83, 53.12, 61.18,63.42) corresponding to graphite lattice reflections of Cu-K∝ photons can be observed at Fig. 2. The particle/grain size of graphite nanoparticles were determined by the X-ray line broadening method using the Scherer equation(from the FWHM of the most intensive peak at $26.7°$) was found out to be about nm.[29]:

Scherer equation

$$\beta = \frac{k\lambda}{D\cos\theta} + \varepsilon \tan\theta \qquad (1)$$

where D is the crystallite size in nanometers, λ is the wavelength of the radiation (1.54056 Å for Cu-K∝ radiation), k is a constant equal to 0.94, β is the peak width at half maximum intensity, and θ is the peak position, ε is lattice strain.

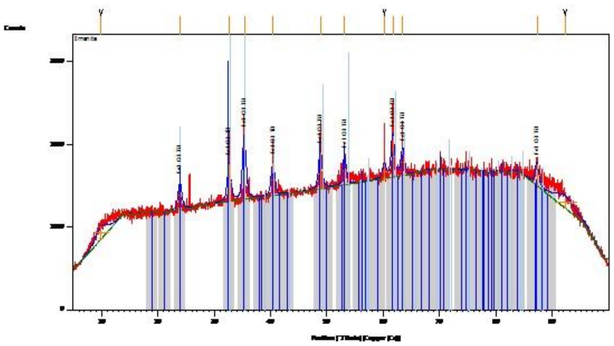

Fig. 2 XRD pattern of ilmenite nanoparticles.

VI. SMALL ANGLE X-RAY SCATTERING (SAXS) ANALYSIS

SAXS is usually applied to tasks of determining spatial sizes for clusters, crystallites, powder and polycrystalline grains, layer thicknesses and superlattice periods. All these values are ranging in the order of magnitude from nanometers to hundreds of nanometers n[30]. The standard procedure of SAXS analysis includes data treatment in three different data ranges [30]. The angular scale was transformed initially into the scale of wave vector magnitudes $q = 4\pi \sin \theta/\lambda$ (with θ being one half of the scattering angle). In the vicinity of the incident beam direction the SAXS curve was broadened; the FWHM (full width at half maximum) of this SAXS peak allowed one to estimate homogeneity sizes in the sample. SAXS data analysis has led to following results as shown in Fig. 3. Ilmenite nanoparticles have appeared to contain grains with particle sizes up to 10–40 nm.

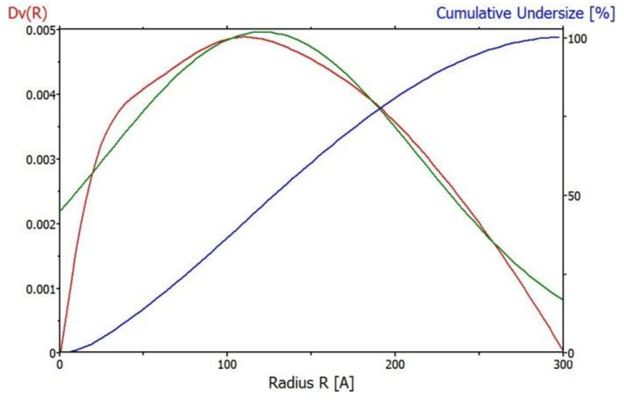

Fig. 3 Particle Size distribution of ilmenite nanoparticles.

VII. STUDY OF MICROWAVE ABSORPTION BY ILMENITE NANOPARTICLES

The test on ilmenite nanoparticles was carried out using microwave bench Klystron Test bench NV9000 and results of microwave absorption were shown in table 1.

Table 1 Microwave absorption data for ilmenite nanoparticles

S no	Frequency (GHz)	Power Mode	VSWR	Reflection coefficient (Γ)	Reflected power (%)	Reflected power (dB)	Absorbed Reflected power (dB)	Return Loss RL
1	8	60	1.13	0.061	11.0	6.60	53.40	24.29
2	9	60	1.13	0.061	11.0	6.60	53.40	24.29
3	10	60	1.14	0.065	12.0	7.20	52.80	23.74
4	11	60	1.16	0.074	13.0	7.80	52.20	22.61
5	12	60	1.21	0.095	33.0	19.8	40.20	20.44

A. VSWR vs Frequency

Measurements were carried out for ilmenite nanoparticles. The results of VSWR measurement for the both sample depending on the frequency are shown in Fig. 4 . The figure shows that the VSWR value depends on particle size of the material. As the particle size decreases VSWR is less, which show that the value of refection from the surface of the material is also less.

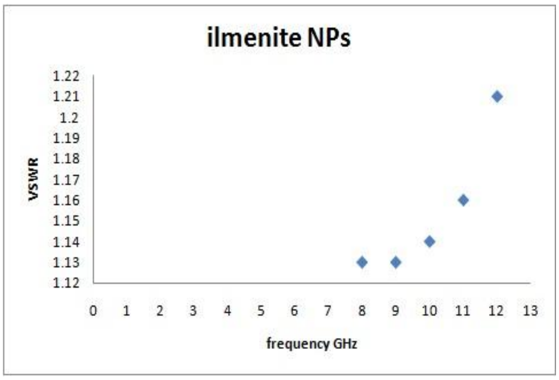

Fig. 4 Graph plotted between VSWR and Frequency.

B. Reflection coefficient vs Frequency

The values of reflection coefficients and frequency are measured using the microwave bench setup (Klystron Test bench NV9000) for different samples in Fig. 5 . The graph drawn between reflection coefficient vs frequency shows that reflection coefficient depends on particle size of the material. The nanoparticles absorbed nearly an average of 80% microwave radiation energy for frequencies ranging from 8GHz to 12GHz.

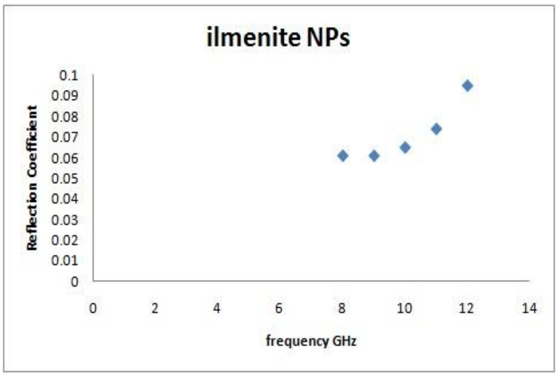

Fig. 5 Graph plotted between Reflection coefficient and Frequency.

C. Return Loss (RL) vs Frequency

The values of reflection coefficient are measured using the microwave bench (Klystron Test bench NV9000) and return loss (RL) is calculated for ilmenite nanoparticles as shown in table 1 and Fig. 6. The graph drawn between return Loss (RL) vs frequency shows that return loss (RL) depends on particle size, crystalline structure and morphology of the material.

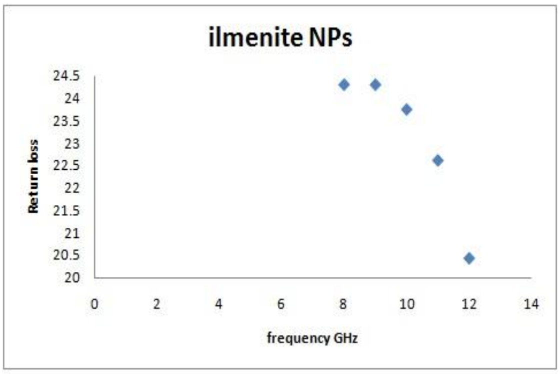

Fig. 6 Graph plotted between Return loss and Frequency

The obtained results showed high levels of absorption of electromagnetic energy by structures based on nanoscale ilmenite particles prospects of using these structures in the design of real RAM.

VIII. DISCUSSION

D. Formation of ilmenite Nanoparticles

Microbes carry out oxidation-reduction reactions in order to obtain energy for growth and cell maintenance. The amount of energy released per electron equivalent of an electron donor oxidized varies considerably from reaction to reaction. Reduction reaction requires electron acceptor type reaction. Full energy reaction ΔG is obtained; the free energy for the donor half reaction is added to the acceptor half reaction [31-33]. The microbes play an important role in many chemical reactions like photosynthesis, fermentation, leaching and oxidation-reduction reactions in Nature. The microbes have an accessory DNA elements present in the cytoplasm called as catabolic plasmids [34]. The plasmids are circular and confer on their host ability to transform or recycle complex, naturally occurring and synthetic molecules.

The biotechnology utilizes microbial environment to produce required chemical reactions. Chemical reactions are taken place due to physical, chemical and biological components interact with each other. These interactions play an important role for molecule or an element to modify its physicochemical form. This process is called transformation. However, the change is not one way, environment too plays the cost in terms of modification of temperature, chemical reaction (pH), conductivity etc.

Biosynthesis of nanoparticles is a promising, green, non-toxic and environmental friendly method. Most of the process can be performed at ambient temperature and pressure and also at neutral pH. After the synthesis of nanoparticles, microorganisms usually still have the ability to reproduce, which indicates that the nanoparticles synthesized by microorganisms show good biocompatibility. Based on these advantages, the synthesis of metallic nanoparticles by microorganisms is receiving increased attention. So, ilmenite nanoparticles were synthesized under microbial conditions. These NPs might be formed due to change in chemical environmental conditions.

E.Absorption of Microwave waves by Ilmenite NPs

Nanostructured RAMs possess enhanced absorbing property due to the nanometer size. The morphology and size of the nanostructured RAMs play very important roles in the microwave return loss (RL) of the nanoparticles and, as the degree or extent of the crystallinity increases, the systematic increment in RL appears. The magnetic-dielectric absorbers of nanocomposite RAMs with suitable dielectric and magnetic properties possess high efficiency because of the complex permittivity and permeability that differ from zero. Nanocomposite of inorganic materials RAMs combines better matched characteristic impedance and improved reflection loss of both materials. For nanostructured ilmenite RAMs, the position of reflectivity peak moves to a lower frequency and the loss factors of composites increases with decrease in particle size of the material . So, there has been more efficient shielding properties had been observed for ilmenite NPs.

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