Iron Oxide Fe_3O_4 Nanoparticles for Electromagnetic Shielding

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Abstract

Magnetic iron oxide nanoparticles (Fe_3O_4) can be dispersed in a supporting material so that the composite can better respond to electromagnetic fields, absorbing a part of their energy. In the discussion here proposed, therefore, we will consider the role of these nanoparticles in the applications for electromagnetic interference (EMI) shielding. Inserted in intrinsically conducting polymers (ICPs) for instance, the nanoparticles are increasing EMI shielding effectiveness of polymer, producing light weight "absorption-type" shields, which are specifically relevant for absorbing microwaves. Composite materials, based on Fe_3O_4 nanoparticles with polypyrrole and polyaniline will be considered in detail. Other materials, such as the recent biochar-based composites, will be discussed too.

Keywords: **Magnetic iron oxide nanoparticles, Fe_3O_4, Magnetite, Electromagnetic interference shielding effectiveness, EMI-SE, Microwaves absorption, Intrinsically conducting polymers, Polypyrrole, Polyaniline, EMI shielding textiles, Biochar.**

Subject Areas: **Materials Science.**

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Introduction

Electromagnetic shielding applications are relevant to protect human health and electronic devices against dangerous effects of electromagnetic radiations (Sparavigna et al., 2005). Metals are usually considered the best materials for electromagnetic shields, due to their peculiar ability in reflecting the electromagnetic waves. In fact, metals or metal-coated materials generally show a very high electromagnetic interference shielding effectiveness EMI-SE, because their high conductivity makes them shield by surface reflection (Henn & Silverman, 1991, Henn & Cribb, 1983). However, metals and metal-coated materials cannot be used as electromagnetic wave absorbers. Materials, such as intrinsically conducting polymers (ICPs) are able to absorb as well as reflect electromagnetic waves, and exhibit certain advantages over metallic materials, such as easy preparation, light weight, good environmental resistance (Soares et al., 2021). The most prominent ICPs for EMI-SE are polypyrrole PPy and polyaniline PANI, where electrical conductivity can have values comparable to those observed for poorly conducting metals and alloy.

Previously, we have considered ICPs for EMI shielding (Avloni et al., 2006), particularly PPy in composites for woven and non-woven fabrics (Avloni et al., 2007, 2008), mentioning the importance of absorption of radiation. In fact, absorption of textiles with PPy is in percentage relevant: it was measured to constitute the 17% for a twill sample, and 15% for a non-woven PPy coated sample, of the total insertion loss. For metallized fabrics it was of 4% (see Avloni et al., 2007, for details). However, the use of specific nanoparticles can relevantly increase the intrinsic EMI absorption mechanism of ICPs, producing in this manner light weight "absorption-type" shielding materials, today specifically investigated to obtain microwave absorption. Due to the use of electromagnetic waves in the GHz range for mobile phone and radar systems, microwave absorbing materials are regarded as very attractive. Here we will consider in particular the magnetic iron oxide nanoparticles of magnetite (Fe_3O_4).

Responding to an external magnetic field, the magnetite nanoparticles are able of absorbing the electromagnetic energy (Ni et al., 2009). As we will deduce from literature, Fe_3O_4 nanoparticles are suitable for obtaining absorbing EMI shielding composites, based on several different supporting materials, ranging from polymers to eco-friendly materials, such as the more recently considered biochar for instance. As several researchers have stressed, the different absorbing mechanisms of nanoparticles and supporting materials are acting synergically in improving the shielding effectiveness.

For what regards the composites with ICPs we will start from the analysis of Fe_3O_4 with PPy, but before, some words about magnetite nanoparticles are necessary.

Fe_3O_4 nanoparticles production

In Blaney, 2007, we can find the detailed description of magnetite (Fe_3O_4). A section of the article explores the bulk properties of it. For the electrical properties, the conductivity ranges from 10^2–10^3 $\Omega^{-1}cm^{-1}$ (Cornell & Schwertmann, 1996). This electrical conductivity is evidencing a semi-conductor behavior, bordering the conductor (metallic) behavior. A semi-metallic behavior is further supported by magnetite relatively low bandgap (0.1 eV) (Cornell & Schwertmann, 1996). The Curie temperature is at 850 K. Below the Curie temperature, magnetite is a ferrimagnetic material (Cornell & Schwertmann, 1996). When the Curie temperature is attained, a superparamagnetic behavior is observed.

If we consider the particles, as the particle size decreases, the behavior tends towards a paramagnetic or superparamagnetic magnetization. Therefore, the decreasing of the particle size reduces ferrimagnetic behavior and enhances superparamagnetic behavior (Blaney, 2007). Also the Curie temperature is affected by the particle size; since the superparamagnetic magnetism is observed at room temperature, the effective Curie temperature of the nanoparticles must be lower than that of the bulk material (Blaney, 2007).

In his article, Lee Blaney gives a detailed discussion of the nanoparticle synthesis techniques. The majority of these techniques falls under two categories: polymer/surfactant assisted precipitation reactions and co-precipitation reactions. One of the surfactant assisted reaction is the reverse micelle method. It is based on a water-in-oil emulsion that generate reverse surfactant micelles. The hydrophilic cores of reverse micelles are nanoreactors for various processes. Metal species can be dissolved into the aqueous phase contained within the reverse micellar cores. In this manner a nanoreactor is obtained, containing the species of interest at the reverse micelle center. Simultaneous production and steric stabilization of the nanoparticles can be realized.

For Fe_3O_4 the following procedure reported by Blaney is that given by Lee et al., 2005, who have detailed a protocol to have nanoparticles of uniform diameters over the 2-10 nm range. The particle size is given by the relative amounts of surfactants, solvents, and polar solvents.

The used surfactant is the dodecylbenzenesulfonate. It is added to an oil (xylene) solution. The emulsion solution is mixed by sonication. An iron solution with 1:2 (molar ratio) of ferrous chloride and ferric nitrate in ethanol is then vigorously stirred into the emulsion solution. In a few seconds, the emulsion becomes transparent and after stirring for 12 hours, the reverse micelle phase, that is the water-in-oil phase, is stabilized. Then a gradual heating of the phase to 90 °C in argon flow ensues. Hydrazine is added to the system and immediately the transparent solution becomes black. Refluxing and centrifugation in ethanol recover the magnetite nanoparticles.

Fe_3O_4 nanoparticles are sometimes mentioned as MIONs (Monocrystalline Iron Oxide Nanoparticles). Dispersed in a supporting material, they are able to respond to an external magnetic field (Tong. et al., 2019).

Fe_3O_4@polypyrrole and EMI absorption

In Deng et al., 2003, we can find how to prepare Fe_3O_4–polypyrrole nanoparticles with core-shell structure. Deng and coworkers prepared a "Fe_3O_4 magnetic fluid" by a precipitation–oxidation method. The starting mixture is of PEG and $FeSO_4 \cdot 7H_2O$ dissolved in water. After the method described in the article, the magnetic fluid was obtained and used for in situ emulsion polymerization in aqueous solution and NaDS. Water and NaDS are placed in a flask, stirred, and then magnetic fluid added and stirred. Then pyrrole is added and all stirred. The polymerization is started by adding $FeCl_3$ dissolved in water and added dropwisely. The polymerization takes place at 0–5 ◦C for 10 hours with stirring. After further procedures, Fe_3O_4–polypyrrole nanoparticles with core-shell structure are obtained. The transmission electron microscopy images of Fe_3O_4 and Fe_3O_4–polypyrrole particles show spherical particles with sizes ranging from 20 to 30 nm and 30 to 40 nm, respectively. The composite has been investigated by IR, UV–visible and X-ray photoelectron spectroscopy (XPS) spectra. In the article by Deng and coworkers, no mention to EMI shielding or to microwave absorption is given.

Also Li et al., 2011, have proposed the synthesis of Fe_3O_4/PPy core/shell nanocomposites, where Fe_3O_4 nanoparticles are used as seeds for in situ polymerization on them. The synthesis of Fe_3O_4/PPy core/shell nanocomposites was made as follow. Fe_3O_4 nanoparticles were dispersed in distilled water by ultrasound treatment to form a dark solution. Then, the pyrrole monomers were slowly inserted in the mixture under ultrasound. Subsequently, to the mixture a $(NH_4)_2S_2O_8$ solution was added dropwisely. In 24 hours, we have the polymerization at room temperature, under constant stirring. The final product was centrifuged, washed and dried. Li and coworkers measured the complex permeability and permittivity of the related composite materials to evaluate their microwave absorption properties. For measurements, the Fe_3O_4/PPy nanocomposites were mixed with paraffin wax sample. The real parts of complex permittivity and permeability represent - Li et al. are explaining - the storage of electric and magnetic energy, while the imaginary parts "symbolize the loss of electric and magnetic energy". "Compared to other microwave absorption materials", the imaginary permittivity is higher (Li et al., 2011). "This phenomenon may result from the PPy shell due to its dielectric loss at high frequencies", and Li and coworkers stress that it is due to the core/shell structure which produces an additional *interface with an interfacial polarization* at the surface of nanoparticles. The resulting higher imaginary part of permittivity means "more dielectric loss and makes more electromagnetic energy transfer into heat energy" (Li et al., 2011). A higher imaginary part of permittivity "could improve the microwave absorption property of the sample", according to Li and coworkers.

In 2020, Liu and Liao have proposed again Fe_3O_4 nanoparticles and polypyrrole for preparing absorption-type electromagnetic interference shields and devices for radar stealth. The researchers observe that the "absorption-type" shielding materials have a specific significance in the protection of humans and electronic devices against electromagnetic radiation which is reflected by metallic surfaces. Therefore, these surfaces are causing a secondary electromagnetic pollution. As already pointed out (Avloni et al., 2008), it is important to obtain an EMI-SE with a high absorption contribution.

To prepare an absorption-type shielding material, as we have previously told, suitable permittivity and permeability are required, accompanied by a finite electrical conductivity. As told by Arora et al., 2014, materials with high dielectric constant like ZnO, SiO_2, TiO_2, $BaTiO_3$, or high magnetic permeability like carbonyl iron, Ni, Co, Fe metals, γ-Fe_2O_3, Fe_3O_4, are used. Like Li et al, 2011, Liu and Liao consider both strategies related to electric and magnetic dipoles. They used Fe_3O_4 nanoparticles with polypyrrole (PPy) coating, fixed onto collagen fibers (CFs).

About the synthesis of $CF_{0.5}Fe_3O_{4x}PPy_y$ composite, Liu and Liao used CFs (0.5 g) and Fe_3O_4 (x g) NPs were immersed in 30 mL of 50 v% ethanol solution, stirring for 1.0 h, so that the CF/Fe_3O_4 composite was obtained by drying. Second, Py monomer (y g) was added to the solution at 3.0 °C for 0.5 h. Third, $FeCl_3 \cdot 6H_2O$ (z g) was dissolved in 10 mL of HCl; the $FeCl_3$ solution served as an initiator of reaction, added dropwisely under continuous stirring. After the next 18.0 h at 3.0°C, the product was filtered, washed and dried. The final powder sample was compressed into tablets of various thicknesses. In this manner, Liu and Liao were able of inserting in the scaffold of the collagen microfibrils, a high loading of "magnetic-loss-activated" Fe_3O_4 nanoparticles separated inside the pores, then coated by a "dielectric-loss-activated shell of conductive polypyrrole (PPy)". Measurements made by researchers show that the incident EMI "energy can be dissipated by dielectric loss, magnetic loss, and interfacial polarization loss simultaneously" (Liu & Liao, 2020).

The hierarchical structure of the assembly of collagen fibers is characterized by pores from the nano- to the microscale, that are producing multiple reflection and scattering of waves. The porosity is therefore improving the attenuation capacity produced by Fe_3O_4 and PPy. The researchers are giving for their composites an EMI-SE value of ~72.0 dB, with an absorption contribution of 85.8%, with high radar-stealth performance (8.2–11.5 GHz).

Let us also add that, as stressed by Liu and Liao, the impedance matching is a determinant feature for absorptive shielding material. In this case, the incident wave is transmitted into the material where it can be absorbed. The matching is determined by permittivity ($\varepsilon_r = \varepsilon' - j\varepsilon''$) and permeability ($\mu_r = \mu' - j\mu''$). Permittivity is a complex quantity with real and imaginary parts. The imaginary part (ε'') is associated with dielectric losses and the real part (ε') related to polarization. Complex permeability has features similar to those of complex permittivity and magnetization, like polarization, can be source of dissipation. Real parts of permittivity (ε') and permeability (μ') refer to the storage capability of electric and magnetic energy, related to the generation of dipoles and to the polarization of the shielding material. The imaginary part (ε'' and μ'') is related to the dissipation of electric and magnetic energy, because of the orientation of dipoles, and other mechanisms, caused by the electromagnetic field.

As told by Li et al., 2017, for Co@C composites, the impedance matching and the microwave attenuation "play key roles in electromagnetic absorption". By means of "moderate electromagnetic parameters", the composite material can have impedance matching together with attenuation ability. "Using magnetic/dielectric composites is considered to be an efficient strategy for achieving excellent electromagnetic absorbing properties" (Li et al., 2017).

Liu and Liao show that the increase in the composite of the presence of Fe_3O_4 nanoparticles NPs improves the magnetic properties, favoring the EMI dissipation through the resonance loss and eddy current loss (Wen, et al., 2011). And also, introducing magnetic Fe_3O_4 NPs "*easily* realized the impedance matching between the shield and free space" (Liu & Liao, 2020). For what concerns the reflection loss RL, Liu and Liao tell that an "RL value smaller than −5.0, −10.0, or −20.0 dB indicated that more than 70.0%, 90.0%, or 99.0% of incident EMWs [electromagnetic waves] are absorbed. The minimum RL values of the five samples were around −5.0 dB with a thickness of 2.0 mm." (Liu & Liao, 2020).

Of course, the absorption of microwaves depends on the sample thickness.

Microwave absorbing materials (MAM)

Adebayo et al., 2020, also stress the importance of new materials for microwave absorption (frequencies between 300 MHz and 300 GHz). For the novel microwave absorbing materials (MAMs), magnetite (Fe_3O_4) is "thoroughly investigated", because of its permittivity and permeability and proper magnetization and Curie temperature (Adebayo et al., 2020). Limiting factors exist, such as the weight and the impedance mismatch, but they can be circumvented passing to the nanoscale, with the creation of hierarchical frameworks, and using magnetite with other lossy materials. The article by Adebayo and coworkers is detailing also the electromagnetic mechanism. Here we just remember that we need an impedance of the absorptive material close to that of vacuum (377 Ω) and a rapidly attenuation of the waves in the material. As previously seen, we need magnetic and dielectric loss ability. The reduction of the impedance mismatch is important, because a high mismatch results in high reflection of waves. For this reason, the design of *multilayer* absorptive materials, which are able of avoiding reflection and absorbing the waves, operating in the bandwidth of microwaves, are investigated. As reported by Adebayo et al., Xu et al., 2015, solved the problem with a multilayer comprising of PANI and Fe_3O_4/PANI. They obtained maximum absorption of about 54 dB.

Permittivity and permeability

Materials are usually classified into conductors, semiconductors and dielectrics. Focusing on dielectric materials, for their applications it is essential to know the dielectric constant, that is the real part of complex permittivity, and the loss tangent at the given operating conditions (Brodie et al., 2015). A dielectric is a material which is polarized under an electric field (dielectric polarization). The polarizability of the material is given by the permittivity, which is a complex number. Together with the magnetic permeability, permittivity is determining the EM field propagation in the material. As shown by Lifšits and Pitaevskij, 1986, in alternating electromagnetic fields, permittivity and permeability are complex quantities with an imaginary part which is always different from zero, according to causality principle and thermodynamics principles.

Starting from Maxwell’s equations, we can explain the dielectric properties of materials. According to Brodie et al., 2015, we have the complex permittivity given by:

$$\varepsilon_c = \varepsilon' - j\varepsilon'' = \varepsilon - j\frac{\sigma}{\omega} \quad ,$$

where we find the real dielectric constant ε , the imaginary part expressed as a "conductivity" σ of the dielectric and the angular frequency ω . About this form of proposing the imaginary part of the permittivity, in all the frequency domain, see please the discussion given in Lifšits and Pitaevskij, 1986, Chapter 9, page 392.

The real part of the permittivity is related to the amount of energy of the electrical field stored in the material. The imaginary part is related to the amount of energy loss. The loss tangent is:

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \quad .$$

As given by Lifšits and Pitaevskij (1986) and Boris Katsenelenbaum, 2006, in his book on high-frequency electrodynamics, the energy dissipation is related to the divergence of Poynting vector. We can obtain the heat produced at a local density rate, being proportional to the imaginary part of permittivity and permeability.

About the complex permittivity and permeability linked to losses in materials, let us consider some different manners in proposing the subject. Let us start from the discussion by Qin et al., 2022, about the dielectric loss mechanisms relevant for electromagnetic wave EMW absorption. The review is providing a comprehensive summary on dielectric mechanisms, including dipolar polarization, defect related polarization, space charge and interfacial polarization and dielectric (conduction) loss.

To have a good EMW absorber, the practice is to increase dielectric and magnetic losses, which are causing EMW dissipation. The dielectric losses are due to different mechanisms: for EMW absorbers tailored in 2–18 GHz frequency range, we have to consider dielectric conductivity and polarization due to the presence of dipoles, interfaces and defects. Other mechanisms, which are ionic, atomic and electronic polarizations are seldom involved because they are relevant in the range of 10^3–10^6 GHz. In the review by Qin and coworkers, it is stressed that composites use magnetic materials, that can provide magnetic loss, and high-conductive materials that provide conductive loss. Then, "magnetic metal and carbonyl iron coupled with high-conductive carbonaceous materials (graphene, carbon nanotube) and conducting polymers are widely explored to create high-performance absorbers" (see Qin et al., 2022, and references therein). Qin and coworkers are mentioning in particular the work by Ding et al., 2019, that "boosted interfacial polarization from multishell TiO_2@Fe_3O_4@PPy heterojunction" to improve microwave absorption MA. To have interfacial polarization, porous or hollow materials are used too. For MA, porous or hollow structure is suitable for materials with high conductive, such as the carbon materials. The analysis of interfacial polarization promotion shows that the use of porous absorbing materials is enhancing interfacial polarization loss (Qin et al., 2022).

The review continues with dipole polarization, distinguishing between displacement polarization and orientation polarization. The polar molecules display dipolar polarization and relaxation processes when alternating EM fields are applied, so that EM energy is dissipated. In the review by Qin and coworkers, it is noted that polar functional groups such as carbonyl and hydroxide radicals "can also act as electric dipoles" relevant for EM energy losses. The "dipolar polarization loss in absorbers stems from the existence of polar molecules and polar functional groups" (Qin et al., 2022). After mentioning the calcination of precursors at high temperature, it is stressed that in many researches the main source of dipolar polarization is in the "functional groups in carbonaceous materials". "Functional groups are rich in most carbonaceous materials including carbon fibers, carbon nanotubes, *biochar*, and graphene" (see Qin et al, 2022, and references therein). Moreover, to promote the dipolar polarization loss, a strategy is that of anchoring functional groups on the surfaces of absorbing materials.

Conduction loss is caused by the EM wave propagation when its energy is converted into charge transport energy. "Conductive loss exists in absorbers with high conductivity", and Qin and coworkers mention again carbon materials, such as graphene, carbon fibers, carbon nanotubes, and the conducting polypyrrole and polyaniline. The problem is that high conductivity turns into a mismatch of impedance, that is good reflection and poor transmission, and consequently reduced absorption. About the conductive loss, two models are used. The first model is the "electron migrating" model. When a charge current is generated by an external EM field, the "electron migrating " part is referring to the free electron propagation in the medium, that is in a nanotube or in the 2D-layer of graphene for instance. The other model is the "electron hopping" model. It refers to the charge transfer between the "components, interfaces and defects" (Qin et al., 2022). This model requires enough filling ratio of absorbing elements. When the conductive network condition is satisfied, we have hopping electrons in the network and the presence of micro-current in it.

Another review we can mention is that by Ganguly et al., 2018, about polymer nanocomposites for EMI shielding. Again, we can find that the presence of electric and magnetic dipoles "can help in absorption of the radiation by the shield". Materials with high permittivity, for instance $BaTiO_3$, are providing electric dipoles, whereas magnetic dipoles can be provided by Fe_3O_4 or other materials with high magnetic permeability. Absorption loss is depending on the product $\sigma\mu$ of conductivity and permeability. When an EMW passes through a medium, the induction of currents in it produces "ohmic losses and heating of the material" (Ganguly et al., 2018). In Ganguly et al., 2018, we can find mentioned the development of a high-performance shielding material in the form of polyaniline (PANI) based nanocomposite and Fe_3O_4 decorated graphite oxide (Singh et al. 2013). EMI-SE is of 26 dB at 15 GHz, with reflection loss of 6 dB. Another interesting work about Fe_3O_4 is that by Hosseini et al., 2011, who coated polythiophene nanofibers with $MnFe_2O_4/Fe_3O_4$ core–shell nanoparticles, synthesized with co-precipitation and in situ polymerization.

Kruželák et al., 2021, consider EMI-SE absorption of rubber composites based on ferrite and carbon fillers (carbon black and nanotubes). We can find in this work a discussion of complex permeability and permittivity too. Real permeability represents magnetic storage capacity, whereas imaginary permeability gives magnetic dissipation or losses. From experimental data, it is seen that the composites filled only with carbon black or carbon nanotubes have low real permeability and negligible imaginary permeability. This fact is displaying a non-magnetic character of carbon-based fillers. The incorporation of ferrite, that has magnetic dipoles, increases of complex permeability. The permeability is depending on "spin precession, domain wall movement, hysteresis loss and eddy current effect" (Kruželák et al., 2021). The hysteresis loss are negligible when low magnetic fields and high frequencies are involved. Spin precession and domain wall movement are connected with resonance phenomena observed in the permeability spectrum. Eddy current effect might also contribute to permeability loss, relevant for small particles.

The real part of the permittivity ε' represents the "electrical charge storage capacity" and can be understood as simulated by micro-capacitors. "Micro-capacitors are formed by particles or aggregates of the fillers that act as electrodes filled with insulating rubber matrix" (Kruželák et al., 2021). Defects of the filler structure act as polarization centers. Therefore, the increase of the presence of micro-capacitors and structural defects produces an increase in real permittivity of composites. The increase of the filler loading reduces the gap between filler particles; this produces an increase of the polarization of the rubber matrix. In the case the concentration of fillers (conductive fillers) reaches the *percolation threshold*, there is the formation of filler conductive paths. The conductive filler networks are acting as dissipating mobile charge carriers too. Actually, several phenomena and related relaxations are influencing the permittivity. "Interfacial and space charge relaxations occur because charge carriers are trapped at the interfaces of heterogeneous composite system" (Kruželák et al., 2021).

Ruiz-Perez et al., 2022, are also considering superparamagnetism in their work about carbon-based radar absorbing material. Ruiz-Perez and coworkers refer that a method to "improve the electromagnetic attenuation response of materials" is based on the insertion of metals or semiconductors to "modulate the electrical conductivity of insulating matrices". But, when the electrical conductivity of the material is high, we have an interface impedance mismatch. This mismatch increases reflectivity and decreases microwave absorption. On the other hand, if we reduce the conductivity by reducing the percentage of conductive fillers in the composite below the percolation threshold, the microwave absorption response turns out to be reduced.

"Several strategies have been addressed to improve the dielectric properties of carbon-based polymer composites, including the hybridization with magnetic metals" (Ruiz-Perez et al., 2022). As previously told, we have also the magnetic materials, which are providing different loss mechanisms, According to Ruiz-Perez and coworkers, "super-paramagnetic materials are the best option for microwave absorber compounds". Accordingly, Fe_3O_4 nanoparticles are interesting for microwave absorbers.

Wang et al., 2021, consider both the dielectric and the magnetic losses, besides the impedance matching condition, in their discussion about the progress of microwave absorption of materials by "magnetic–dielectric synergy". Under the "polarization loss", we can find that the loss is mainly due to dipole polarization and interface polarization. The dipoles are linked to "active sites", given by defects and surface functional groups. "When the carriers accumulated at the interfaces", we have generated the interfacial relaxation, which can dissipate the incident EM energy. Equivalent circuit models have been proposed for interfacial polarization. The proposed example is the peak at about 14.1 GHz for the imaginary part of the permittivity in the case of Fe_3O_4/Carbon-Nanotubes hybrids, corresponding to the heterojunction capacitor produced by the interface between Fe_3O_4 and nanotubes. About the "conduction loss", Wang and coworkers are mentioning the carbon-based materials and some polymer materials. Considering the "transmission line theory", the conductivity with electronic transport is proportional to the values of ε'' (Wang et al., 2021, Cao et al. 2012): in alternating EM fields, "some electrons" can "move directly", other electrons can "jump across the interface".

About the magnetic loss, when an EM wave enters the material, a dynamic magnetization process is created which is consuming energy. Thus, also in this case, the magnetic permeability is expressed as $\mu = \mu' - j\mu''$, where real μ' and imaginary μ'' parts of permeability are depending on frequency. Wang and coworkers note that the frequency range can be subdivided into some main regions. In the low frequency region, less than 10^6 Hz, the values of μ' and μ'' change little. In the frequency range (10^6–10^8 Hz), μ' is rapidly decreasing, whereas μ'' has a peak, mainly due to the resonance of magnetic domain walls. In the range 10^8 –10^{10} Hz, natural resonance is occurring. When the frequency is greater than 10^{10} Hz, we have the ferromagnetic resonance. "Magnetic loss mainly includes hysteresis loss, domain wall resonance, natural resonance, ferromagnetic resonance and eddy current loss" (Wang et al., 2021). The natural resonance is that which appears at the frequency of precession of magnetic moments in the material (Sabu et al., 2012).

Fe_3O_4@Graphite

Wang et al., 2022, have proposed Fe_3O_4–Graphite composites as MAMs. In their approach, electromagnetic features and impedance matching were regulated by the graphite percentage. The optimal ratio for bimodal performance is 3:10 (Fe_3O_4–2PG, Pre-treated Graphite). With a thickness of 4 mm, the minimum reflection loss (RL_{min}) in C-band (4–8 GHz) was measured as −40.6 dB, and RL_{min} in Ku-band (12–18 GHz) was −29.82 dB. The performance was attributed to the “synergistic effects and interfacial polarization between Fe_3O_4 nanoparticles and graphite”. The composite materials have electromagnetic absorption peaks in both the C-band and Ku-band, therefore with relevance for 5 G technology and the shielding absorption of C-band radar waves (Wang et al., 2022).

Here the point of view by Wang and coworkers about Fe_3O_4 nanoparticles. "Among various electromagnetic microwave absorption materials”, these nanoparticles have the advantages of being environmental friendly material, abundant, and with controllable preparation technology. “Fe_3O_4 nanoparticles *as dielectric loss materials can produce dielectric loss characteristics* and improve the microwave absorption (MA) properties of materials”. Moreover, the material’s impedance matching can reduce reflection of EM waves. Wang et al., 2022, provides several references to research studies about composites of Fe_3O_4 with carbon-based materials (see references therein). For what regards graphite, it “is one of the early aircraft skin inter-layer-filled absorbing materials. Due to the stacked structure of graphite sheets, it has specific absorbing properties” (Wang et al., 2022, Peng, et al., 2018). Graphite has a low density, accompanied by a high specific surface area, with good electrical conductivity. Combined with Fe_3O_4 nanoparticles, the material is a composite which both magnetic loss and electrical loss, with improved impedance matching characteristics. "Finally, the reflection loss of Fe_3O_4 generally occurs at a lower frequency region, and the reflection loss of graphite is usually located in the high-frequency region" (Wang et al., 2022). In this manner, the interval of absortion is expanded.

"The magnetic loss of materials mainly comes from the domain wall resonance, eddy current effect, hysteresis loss, natural resonance, and exchange resonance" (Wang et al., 2022), but in the case of Fe_3O_4, domain wall resonance and hysteresis losses are not the principal mechanism of magnetic loss in Fe_3O_4–PG. The magnetic loss is dominated by natural resonance in the range of 2–10 GHz, and by eddy current loss in the range of 10–18 GHz.

Nanoparticles and textiles

Let us return to textile applications and ICPs.

Saini et al., in 2012, proposed composites using polyaniline-coated cotton fabrics, incorporating in the coating some dielectric and magnetic nanoparticles, that is nanoparticles of $BaTiO_3$ (15–25 nm) and Fe_3O_4 (25–40 nm) into a poly(aniline) (PANI) matrix. The researchers used a surfactant, dodecylbenzenesulfonate (DBSA), to have a uniform dispersion of inorganic fillers, $BaTiO_3$ or Fe_3O_4, without affecting their intrinsic properties.

In a typical reaction, - Saini and coworkers explain - 0.3 mol of dopant DBSA was homogenized with 1.0 L of water. Then, 0.1 mol of aniline AN monomer was added and homogenized further to form AN-DBSA micelles. The cotton fabric was placed in a reactor with the AN-DBSA solution. The polymerization was produced by dropwise addition of aqueous ammonium peroxydisulfate (0.1 mol) under continuous shaking for 6 hours. A thick coating of PANI was formed over the fabric. After washing and drying the fabric, the coating process was repeated. In the similar manner, fabrics with $BaTiO_3$ and Fe_3O_4 nanoparticles were prepared by adding specific amounts of $BaTiO_3/Fe_3O_4$ after addition of DBSA, keeping the same all other ingredients and reaction conditions. As discussed by Saini et al., the uniform dispersion of nanoparticles within the polymer is fundamental. For relatively high density of ferrites/titanate fillers, it can result a poor dispersion of fillers within the polymer matrix. "Such phase separation and microscopic heterogeneity result in poor electromagnetic properties" (Saini et al., 2012). "However, aqueous solutions of amphiphilic molecules like DBSA possess enough viscosity to counteract the agglomeration and settling tendencies of the above fillers" (Saini et al., 2012).

Dielectric measurements give that the incorporation of $BaTiO_3$ is increasing the dielectric properties of PANI, the same from magnetization measurements that show the relevance of the incorporation of Fe_3O_4 in the improvement of magnetic properties. According to the researchers, the use of nanoparticles is producing "better matching of input impedance, reduction of skin depth, and additional dielectric/magnetic losses." The consequent *specific shielding effectiveness* value of 17–20 dB cm^3/g is a proper indication that these fabrics are suitable for obtaining microwave-shielding materials, besides displaying good antistatic features.

Recently, Yörük et al., 2021, deposited PANI with nanoparticles onto polypropylene nonwoven, so that "one of the most industrially prominent" nonwoven was used to create an EMI shield. The magnetic nanoparticles used by the researchers were Fe_3O_4, $CoFe_2O_4$, and $MnFe_2O_4$. "Accordingly, the double-coated PP/$MnFe_2O_4$/PAn [polyaniline] composite showed the highest absorption-dominant attenuation (94.2% at 2.11 GHz) with a 12.4 dB EMSE value" (Yörük et al., 2021). Hollow magnetic Fe_3O_4 nanospheres have been also used for flexible nonwoven fabrics, by Zheng et al., 2023. The Fe_3O_4/MXene, that is a transition metal carbide, composite fabric has an EMI-SE of 33.28 dB. The EMI shielding mechanism is "*tunable according to the loaded active materials*. Thus, the mechanism can alternate from absorption to reflection" (Zheng et al., 2023). To have a lighter weight, the nanoparticles are hollow, a strategy already pursued in the past.

Fe_3O_4/CarbonBlack@Epoxy-Resin-Matrices

Fallah et al., 2022, studied the EMI shielding properties of polymer nanocomposites possessing different weight percentages of Fe_3O_4 nanoparticles and of “cost-effective” carbon black nanoparticles (CBNs). Nanocomposites were prepared by mixing and casting. EMI-SE was investigated in the frequency range of 8.2 ~ 12.4 GHz. The maximum SE was 36.6 dB at 8.2 GHz for a composite with weight percentage of 15% Fe_3O_4 and 50% CBN (0.7 mm thickness).

About composites, it is told that an epoxy resin (EI-403, Mokarrar Engineering Material Co. Nanomaterials) was the matrix, spherical nanoparticles CBN and Fe_3O_4 were the fillers. Fallah and coworkers tell that “the high electrical conductivity of Fe_3O_4 and CBN is one of the main reasons” for the high EMI-SE observed for their composites. However, “the distribution of CBN is also significant. The uniform distribution of Fe_3O_4 and CBN particles in the matrix established a conducting network”, which is increasing EMI-SE (Fallah et al., 2022). Moreover, “the proper combination of electrical and magnetic losses causes excellent wave absorption” (Fallah et al., 2022).

Film depositions

In the form of magnetic films, the electromagnetic and microwave absorption properties of Fe_3O_4 have been investigated. Wei et al., 2007, used Fe_3O_4 magnetic films plated on hollow glass spheres, to obtain spheres–wax composites. Wei and coworkers observed an improvement of dielectric and magnetic losses, as the volume fraction of the magnetic spheres in the composites increased. The researchers concluded that "the microwave absorbing properties were mainly due to the magnetic loss" (Wei et al., 2007). Fe_3O_4 films on hollow spheres or other low density materials is a good solution to substitute the conventional absorbing materials, ferrites for instance, which have good absorbing properties but are too heavy.

To solve the same problem, Qiang et al., 2010, used carbon fibers as a substrate with low density but high strength, and good electrical properties. Therefore, the "composites of carbon fibers and magnetic materials" have a lighter weight accompanied by good conductivity and strength. As stressed by Qiang and coworkers, Fe_3O_4 is exhibiting "excellent microwave absorption property and have been widely used in many fields".

Li et al., 2013, proposed electroless Ni-Fe_3O_4 composite plating on polyester fabric modified with 3-aminopropyltrimethoxysilane. The EMI-SE of Ni-Fe_3O_4 plated polyester fabric were evaluated. With a Ni-Fe_3O_4 weight on the treated fabric of 32.90 g/m^2, the EMI SE arrives to 15–20 dB for frequencies in the range from 8 to 18 GHz. The results indicate an EMI shielding use of the material (Li et al., 2013).

Paramagnetism and magnetocaloric effect

According to Wei et al., 2012, Fe_3O_4 nanoparticles have been intensively investigated because of their superparamagnetic behavior and for their low Curie temperature. The nanoparticles are also non-toxic and biocompatible, and therefore have several biomedical applications. In the superparamagnetism, and in the absence of an external magnetic field, the magnetization appears to be zero, when averaged on a time longer than Néel relaxation time. If an external magnetic field is applied, nanoparticles behave as paramagnets, with a magnetic susceptibility which is larger than that of paramagnets. Linked to the paramagnetism, we find the magnetocaloric effect. This effect is the adiabatic temperature change of a material upon application of a magnetic field (McMichael et al., 1991).

"By grouping spins together in superparamagnetic clusters", McMichael and coworkers explain, magnetic moments are easily aligned in paramagnetic systems. "For certain ranges of field, temperature and cluster size, the entropy of the spins is more easily changed by application of a field" (McMichael et al., 1991). Magnetocaloric effect is therefore a heating or cooling of a material due to the coupling between the magnetic moments and external magnetic field. In the following Section, we will find that the magnetocaloric effect has been involved in new materials based on biochar.

Black magnetic biochar

Biochar is the black fine-grained residue of the pyrolysis of biomass. It is the product of a thermochemical decomposition at moderate temperatures under oxygen-limiting conditions. This residue has a pore structure with large specific surface area, due to the presence of a mesoporous framework in it. For this reason, biochar can be used for producing shape-stabilized phase change materials SSPCMs (Sparavigna, 2022). The biochar mesoporous framework can encapsulate the liquid phase of PCMs, that are substances absorbing and releasing thermal energy at phase transitions, overcoming the leakage problem of them. One of these SSPCMs is a "black magnetic biochar".

Biochar has several applications. Its main use is for soil emendation, but is can also be used as a filler in polymers and concrete (Brassard et al., 2019, Ok et al., 2015, Bartoli et al., 2020, 2022, Ziegler et al., 2017, Lepak-Kuc et al., 2021, Suarez-Riera et al., 2022), even for electromagnetic shielding (see Appendix). In the recent review by Liang et al., 2022, about phase-change materials, a section of the article is devoted to the use of biochar-based composite PCMs for the storage of solar energy, in the form of thermal energy. Among the reported literature, we can find the article by Yang et al., 2019. The researchers used poplar wood powder to obtain carbonized wood powder (CWF), and then synthesized CWF-PCMs.

Fe_3O_4 nanoparticles have been introduced to prepare a composite able to improve the solar thermal efficiency of CWF-PCMs, because the Fe_3O_4 nanoparticles are adding - as told by Liang et al. - a "black appearance" to biochar so that the absorbed sunlight is increased.

Yang et al., 2019, stress that the composite is a "magnetic wood-based composite phase change material". The researchers used Fe_3O_4 nanoparticles and melted 1-tetradecanol mixed together in weight ratios of 1:100, 2:100, 5:100, and 8:100. The Fe_3O_4 nanoparticles provide magnetic property to the composite. Due to the magnetothermal effect, the composite "can be heated under an alternating magnetic field". The magnetic wood-based composite PCM has a latent heat as large as 179 J/g.

Yang and coworkers do not investigate the properties of the composite related to EMI shielding. This has been made by Shen et al., 2022, who have produced biomass-based carbon aerogels, used as porous supporting material to encapsulate magnetic Fe_3O_4@polyethylene glycol by vacuum impregnation. The researchers tell that they have obtained "excellent thermal storage capability and satisfactory EMI shielding effectiveness (SE)". "With addition of 7 wt% Fe_3O_4," one of the PCMs achieves EMI SE of 53.83 dB. (Shen et al., 2022).

Lignin and graphene oxide (GO) are used by Yang et al.; GO has good mechanical behavior and the electrical conductivity. The lignin@GO suspensions were obtained using several ratios of lignin and GO by stirring and sonication. The lignin@GO suspensions were freeze-dried and thermal annealed to have LG aerogels. Then, LGs were impregnated by pure PEG under vacuum LGs were also impregnated in Fe3O4@PEG with different mass ratios of Fe_3O_4.

Of course, we could also use the Fe_3O_4/PPy core/shell nanoparticles, those proposed by Li et al., 2011, or those by Deng et al., 2003, in the paraffin wax and stabilize them in the macropores of biochar.

Biochar and other microwave absorption materials

A different approach is that proposed by Chen et al., 2023. The researchers are disclosing a facile fabrication of biochar absorbers for enhanced microwave absorption, based on Fe_3O_4 nanoparticles. According to the researchers, the "biochar microwave absorption (MA) materials are attracting significant attention due to their sustainability and cost-effective features". However, to have a significant MA performance, it is necessary to load the biochar with magnetic nanoparticles (NPs). The used approach is based on biotemplates, with Spirulina (SP) cells loaded with Fe_3O_4 NPs.

This biochar is not alone, and Chen and coworkers are reporting several studies on biochar absorbers, which "have gained particular attention due to their unique advantages, including porous structures, superior dielectric loss, low cost, and robust properties" (Zhao et al., 2019, Guan et al., 2021, Natalio et al., 2020). EMI-SE is based on the major mechanisms of reflection, multireflection and absorption. For electromagnetic absorption, it is required the "synergistic effect" between electric and/or magnetic dipoles of the material. Carbon-based materials loaded with metal oxide nanoparticles possessing a high dielectric constant, have been investigated for EMI absorption, but - as stressed by Chen et al. - to improve the magnetic loss of absorbers, the loading of magnetic nanoparticles "not only optimize the impedance matching, but also enhance the overall MA [microwave absorption] performances via synergistic effects of both magnetic and dielectric losses". And therefore we can find the biochar composites (Jute/Fe_3O_4, Coconut shell/FeOx, Pine nut shell/NiO, Glucose/Fe_3O_4, Rice husk/Co, Eggplant peal/Ni), fabricated as biochar absorbers with loading of magnetic NPs (Fang et al., 2017, Liu et al., 2019, Wang et al., 2019, Wang et al., 2022).

Yin et al, 2021, prepared cotton-derived carbon fibers and hollow Fe_3O_4/CoFe hybrids via a two-stage method of hydrothermal and calcination. These hybrids are defined by Yin and coworkers as *biochar* and are proposed as novel low-frequency microwave absorbers. Because of the combined effect of magnetic and dielectric losses, the ternary hybrid possesses "outstanding low-frequency microwave absorbing capacity". The maximal reflection loss values can reach about 40.15 dB. Yin and coworkers claim that the biochar "exhibits huge potential in electromagnetic absorption owing to its rich structures, *splendid* performance, light weight, extensive source, pro-environment and low cost".

Among the references given by Yin et al. we find literature where "biochar-based MAMs present *wonderful* microwave absorbing performance in relatively high-frequency range," so that practicability of biochar for microwave absorption is indicated (Yin et al., 2021). Lou et al., 2018, are displaying the synthesis of magnetic wood fiber boards with electromagnetic wave absorbing properties; in Lv et al., 2016, the article is proposing bacterial cellulose functionalized by sputtering with copper (Cu) and Al_2O_3 to endow it with EMI shielding properties (EMI-SE 65.3 dB); and Marins et al., 2013, are proposing flexible magnetic membranes "with high proportion of magnetite", prepared by impregnation of bacterial cellulose pellicles with ferric chloride, then followed "by reduction with sodium bisulfite and alkaline treatment for magnetite precipitation" (Marins et al., 2013). Microwave properties of the pellicles were investigated in the X-band (8.2 to 12.4 GHz), and the researchers concluded "a potential application as microwave absorber materials" (Marins et al., 2013).

Other reference given by Yin et al, 2021, are reporting a variety of biomass-based carbon materials being "hierarchically porous carbons (HPCs) with excellent microwave absorption (MA) performance" (Wu et al., 2018), with "embedded alkaline metal elements" which are those inside the materials from which biochar is derived. Also carbon foams, with "excellent electromagnetic wave absorption performance", are mentioned (Zhou et al., 2019).

Yin et al, 2021, are also referring to the work by Wang et al., 2018. Materials are based on loofah sponge as hierarchical porous carbon precursors and ferric nitrate as magnetic precursor. "During the carbonization process", the structure of loofah sponge turned into "interconnected networks with hierarchical porous structures, and the precursor ferric nitrate converts into magnetic Fe_3O_4@Fe nanoparticles" (Wang et al., 2018). "As expected", the resulting porous carbon/Fe_3O_4@Fe composites "exhibit outstanding MA performance" (Wang et al., 2018). Wang and coworkers are giving minimum reflection loss (RL) of −49.6 dB with a thickness of 2 mm.

To the above-mentioned works let us add Pan et al., 2022, who are proposing a wood-based composite, consisting in a multilayer structure having a positive conductance gradient and negative magnetic gradient, so that the electromagnetic waves are trapped to underwent a sequence of absorption, reflection and reabsorption, with an additional interfacial polarization loss-induced absorption process. The electromagnetic shielding of the composite can be up to 94.73 dB ranging from 300 kHz to 3.0 GHz.

Since we are talking about wood, let us consider that its lignin is also involved in EMI shielding by Hu et al., 2021. Hu and coworkers prepared “flexible lignin-based electromagnetic shielding polyurethane (FeCLPU) with *excellent* properties”. Lignin was used with carbon nanotubes (CNTs) and Fe_3O_4 nanoparticles in polyurethane “to improve electromagnetic shielding properties” (Hu et al., 2021). The KH550 coupling agent was used for Fe_3O_4. The modified nanoparticles were mixed with lignin, polyethylene glycol (PEG), hexamethylene diisocyanate (HDI) and CNTs to synthesize FeCLPU. “When the concentrations of both Fe_3O_4

and CNT were 10% and the lignin content was 15%, the maximum EMI SE reached 37.5 dB" (Hu et al., 2021).

Fe_3O_4@Honeycomb

Gao et al., 2018, considered honeycomb-like carbonaceous composites to enhance the performance of Fe_3O_4 based absorbing materials. In the introduction of their article, Gao and coworkers tell that the traditional classification of microwave-absorbing materials is based on two classes, one of the magnetic loss materials (Fe, Co, Ni, magnetic alloys), the other of the dielectric loss materials (CuS, SiC, MnO_2, conducting polymers). Carbon nanotubes, filaments and fibers, such as the chemically derived graphene have also considered for EM absorbers. "However, most of these composites still have many disadvantages", and Gao and coworkers mentioned them. For instance, carbon nanotubes and graphene "have a tendency to undergo restacking and aggregation during the preparation process" (Gao et al., 2018), and this could influence the final properties of the material. Moreover, the cost of materials and processing is rather relevant.

Biochar, "widely applied in the capture of carbon dioxide, battery anodes, adsorption of organic pollutants and supercapacitor electrode materials" (Gao et al., 2018), has several proper features. Gao and coworkers are mentioning that biochar possesses intrinsic porosity with large specific surface areas and oxygen-containing functional groups, which can facilitate the formation of chemical bonds with functional groups of modified materials. Moreover, biochar can be activated with KOH so that a considerable number of mesopores is formed inside the material. "At this point, if biochar materials were used as microwave absorbers, microwave energy would be effectively attenuated by *interfacial polarization relaxation loss* owing to the strengthened interfacial polarization induced by solid–void interfaces" (Gao et al., 2018). For this reason, biochar can be considered "promising" candidate for proper absorbers. Gao and coworkers are mentioning the work by Qiu et al., 2017, who used walnut shell-derived nano-porous carbon to produce a microwave absorber based on dielectric loss. The magnetic loss mechanism was not involved.

As stressed by Gao et al., the oxygen-containing functional groups resident on the surface of biochar can be used to modify, by means of a magnetic material, the "impedance matching, which could distinctly improve the microwave absorption performance" (Gao et al., 2018). Gao and coworkers consider the Fe_3O_4 nanoparticles because of their chemical and thermal stability and proper magnetization features. Then, the researchers prepared "a new type of honeycomb-like carbonaceous composite", with a tailored porous framework with Fe_3O_4 NPs, framework obtained from walnut shells. These shells were subjected to two carbonization processes, one at 400 °C, the second at 600 °C after etching with KOH. Then, the porous carbonaceous framework was decorated with Fe_3O_4 nanoparticles NPs. The microwave analysis, shows a "synergistic effect between the porous architectures and magnetic species" (Gao et al., 2018).

The final material, obtained after washing and drying, was named WPC-600 and Gao and coworkers compared it with WC-600 (without etching). WPC-600 and $FeCl_3 \cdot 6H_2O$ were mixed in ethylene glycol by ultrasonication. NaOH and $N_2H_4 \cdot H_2O$ were added and the mixture stirred. After a thermal treatment at 200 °C for 12 h, the resulting black product was washed. The prodct is named Fe_3O_4/WPC-600. "Under solvothermal treatment, Fe_3O_4 nanocrystals were nucleated in situ and grown on the surface of WPC-600, which led to the formation of Fe3O4/WPC-600" (Gao et al., 2018).

Gao and coworkers measured the complex permittivity and permeability of the composite. After the discussion of the related features, the researchers are also remarking that, according to theory, "a higher permittivity means a higher ability to store and dissipate electrical energy, and hence the sample displays better microwave absorption performance. Nevertheless, if the permittivity is much higher than the permeability ... this will result in poor impedance matching" (Gao et al., 2018). Impedance matching means values of permittivity and permeability able to "make electromagnetic waves enter the interior of materials". Measurements tell that WC-600 possesses a high permittivity but, because of a poor impedance matching, it is exhibiting a "poor microwave absorption performance". Gao and coworkers are also mentioning the Maxwell-Garnett theory about composites. "After decoration with Fe_3O_4 nanoparticles", the ε' values of the composite Fe_3O_4/WPC-600 increased. The higher ε' values "might have been due to *space charge polarization* because of the heterogeneity of the composite material" (Gao et al., 2018). The decoration of the surface of porous carbon with Fe_3O_4 nanoparticles generates more space charge and accumulation at the interface, and this contributes to higher microwave absorption in the composite. About permeability μ' and μ'' values, for Fe_3O_4/WPC-600, broad resonance peaks are observed over the whole frequency range (2.0–18.0 GHz). The peaks are attributed to the natural resonance of Fe_3O_4.

Gao and coworkers observe that WPC-600 has a higher dielectric loss tangent with respect WC-600. It means that the KOH etching has improved the microwave absorption because of an increase in the dielectric loss. And the dielectric loss has been increased by numerous defects and mesopores in WPC-600. And the dielectric loss tangent "became much higher after WPC-600 was uniformly decorated with Fe_3O_4 nanoparticles" (Gao et al., 2018). Therefore, the introduction of nanoparticles increased the number of "heterogeneous interfaces, which led to interfacial polarization, dipole polarization and stronger contact relaxation at the interfaces" (Gao et al., 2018). Gao and coworkers mention the Maxwell–Wagner effect, which is appearing in heterogeneous dielectrics, because at interfaces, charges accumulate due to the different dielectric constants and conductivities of the two media. The net result is a polarization.

Let us add that walnut shells have been used also by Li et al., 2020, decorated by Fe_3O_4. And Jia et al., 2021, used Fe_3O_4/α-Fe decorated porous carbon-based composites to adjust impedance matching.

Preparing Fe_3O_4@Biochar

We have found that, in the case of the biochar as shape-stabilizer material for PCMs, Fe_3O_4 nanoparticles have been inserted in the fluid PCM then impregnated in biochar. However, according to the previously proposed literature, the main approach for EMI shielding seems being that of using precursors for the creation of magnetic nanoparticles. Here, let us propose also the method we find in Li et al., 2016, to have Fe_3O_4@Biochar. The spongy pomelo pericarp is used for biochar, grounded into fine powders. Biochar of 1.0 g is mixed with 5.22 g $Fe(NO_3)_3$-$9H_2O$ in 50 mL water. After 1 hour of intense stirring, the mixture is desiccated and dried for 12 hours. Then, it is put into a tube furnace and heated in flowing N_2, and the product is denoted as Fe_3O_4 NP/C (see all details in the article by Li et al., 2016). In the case the as-prepared biochar of 1.0 g is soaked with 1 M nitric acid, the acid-treated carbon (AC) is composited to have Fe_3O_4 NP/AC. In the Fe_3O_4@Biochar, "the mineral substances of uniformly distributed KCl and $CaCO_3$ in the biochar play an important role in enhancing the electrochemical performance of the composite", so that Li et al., 2016, are proposing this composite for anode materials for Li-ion batteries.

A review

As in many other cases, we are facing today a strong increase of the number of publications about new composite materials. This is true also for composites based on the magnetite nanoparticles that we have here discussed. Searching by means of Scholar - fe3o4 nanoparticles electromagnetic shielding - about 21,100 results are proposed. Then, the references here given could appear as "rari nantes in gurgite vasto" (Virgil, Aeneid, I, 188), sparse elements in the immense sea. To navigate this sea, my compass was that of considering the beneficial effect that Fe_3O_4 nanoparticles can have on some materials, such as ICPs, which are already interesting for EMI shielding by themselves. Also the new composites based on biochar are proposed. Biochar is remarkable for its hierarchical porous structure, so that bare or core/shell nanoparticles can be easily inserted in its macropores, enhancing biochar intrinsic EMI shielding features. Of course the review can be expanded to more cases, but there is a key obstacle which is hampering any discussion about EMI shielding in composite materials. It is the fact that experimental results are proposed in rather different manners, turning them into data which are difficult to compare.

Appendix: biochar@concrete

About biochar, we have mentioned the fact that it is compatible with concrete. A recent review about the use of biochar in concrete (Singhal, 2022), tells that biochar is an efficient absorber for EM radiations, besides allowing "buildings to be turned into carbon sinks". Singhal mentions the works by Yasir et al., 2020, who studied shielding properties of cement composites with commercial biochar. At 10 GHz, SE of plain cement was almost 5 dB; for samples with 14 wt.% biochar, SE was of 11 dB, and of 15 dB with 18 wt.% (4-mm-thick samples) (Yasir et al., 2020). Savi et al. (2020) used biochar derived from sewage sludge obtaining SE around 10 dB in the presence of 20% biochar (Savi et al., 2020).

Since our discussion is about the role of magnetite in electromagnetic shielding, let us consider also the work by Das et al., 2022. The study focused on developing EMI shielding cement-based concrete; a maximum SE - around three times than of normal concrete with a similar range of compressive strength - was achieved in conductive concrete with magnetite and graphite aggregates and 2% steel fibres.

For what is regarding the use of biochar in different matrices (included concrete) for EMI-SE evaluation, see please Torsello et al., 2021. Torsello and coworkers reported microwave shielding efficiency in the frequency range 100 MHz–8 GHz. Samples have thickness of 10 and 30 mm. In case of biochar as a filler, as told by Torsello and coworkers, "several aspects intervene due to its intrinsically disordered nature and to the large range of graphitization degrees that can be obtained" in preparing the material; however, "the main contributions to EM shielding in such materials are expected to stem from migration and hopping conductance" (Torsello et al. mentioning Cao et al., 2010).

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