Tomas Bata University in Zlín

Doctoral Thesis-Summary

Preparation and characterization of advanced spinel ferrite nanocomposites for electromagnetic applications

Příprava a charakterizace pokročilých spinel feritových nanokompozitů pro elektromagnetické aplikace

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DEDICATION

This doctoral dissertation is devoted to my parents (Mrs. Sudesh Deswal and Mr. Rajesh Deswal), and my family.

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ABSTRACT

The recent developments in electronic and information transmission technology, has upgraded the life of society. Different technologies associated with electromagnetic (EM) wave such as fifth generation (5G) communication has significantly improved and enhanced people's lives. However, advancement in communication and electronic devices have also increased the EM intereference (EMI) which deterioties the functionality of electronic devices and also influences human-health.

To minimize the EMI, Polymer nanocomposites are a potential candidate to be utillized as an EMI shielding material owing to the flexibility, mechanical properties, ease processing, light-weight and other tailored properties. However, polymer itself is non-magnetic and therefore limiting their application for EMI shielding. A high-performance EM wave absorber should possess strong dielectric and magnetic loss. Recently, spinel ferrites (MFe_2O_4, M = Zn, Co etc.) have drawn significant attention due to their considerable magnetic properties and chemical stability, to be used as a filler inside the polymer composites for EMI shielding application. Moreover, secondary filler such as graphite and reduced graphene oxide are also ideal choice to be utilized as a conducting filler.

In this thesis, various spinel ferrite nanoparticles were synthesized using sonochemical approach. Further, the synthesized spinel ferrite nanoparticles were incorporated along with the conducting filler such as commercially available graphite or synthesized reduced graphene oxide (rGO) in thermoplastic polyurethane (TPU) polymer matrix. This work was focussed on the development of the various polymer nanocomposite systems (a) Zn^{2+} substituted cobalt ferrite nanoparticles along with reduced graphene oxide in TPU matrix (b) Cu^{2+} substituted cobalt ferrite nanoparticles with reduced graphene oxide in TPU matrix (c) optimization of wt% of the conducting graphite and magnetic cobalt ferrite nanoparticles in TPU matrix. Furthermore, the developed nanocomposites were utilized for EMI shielding applications. The EM parameters such as complex permittivity and permeability, dielectric losses, magnetic losses, electrical conductivity were also investigated to study their dependence on the EMI shielding mechanism of the developed polymer nanocomposites. Impedance matching coefficient and attenuation constant helped in predicting the EMI shielding mechanism of the prepared polymer nanocomposites.

ABSTRAKT

Nedávný vývoj v oblasti elektronických technologií a technologií přenosu informací zlepšil život společnosti. Různé technologie spojené s elektromagnetickými (EM) vlnami, jako je komunikace páté generace (5G), výrazně zlepšily a zlepšily životy lidí. Pokrok v oblasti komunikace a elektronických zařízení však také zvýšil elektromagnetickou interferenci (EMI), která zhoršuje funkčnost elektronických zařízení a také ovlivňuje lidské zdraví.

Aby se minimalizovalo EMI, jsou polymerní nanokompozity potenciálním kandidátem na použití jako stínící materiál proti EMI díky flexibilitě, mechanickým vlastnostem, snadnému zpracování, nízké hmotnosti a dalším přizpůsobeným vlastnostem. Samotný polymer je však nemagnetický, a proto omezuje jeho použití pro stínění EMI. Vysoce výkonný absorbér EM vln by měl mít silné dielektrické a magnetické ztráty. V poslední době přitahují značnou pozornost spinelové ferity (MFe_2O_4, M = Zn, Co atd.) díky svým značným magnetickým vlastnostem a chemické stabilitě, které se používají jako plnivo uvnitř polymerních kompozitů pro aplikace stínění EMI. Navíc sekundární plnivo, jako je grafit a redukovaný oxid grafenu, jsou také ideální volbou pro použití jako vodivé plnivo.

V této práci byly pomocí sonochemického přístupu syntetizovány různé spinelové feritové nanočástice. Dále byly syntetizované nanočástice spinelového feritu začleněny spolu s vodivým plnivem, jako je komerčně dostupný grafit nebo syntetizovaný redukovaný oxid grafenu (rGO) v termoplastické polyuretanové (TPU) polymerní matrici. Tato práce byla zaměřena na vývoj různých polymerních nanokompozitních systémů (a) Zn^{2+} substituované nanočástice kobaltového feritu spolu s redukovaným oxidem grafenu v TPU matrici (b) Cu^{2+} substituované kobalt feritové nanočástice s redukovaným oxidem grafenu v TPU matrici (c) optimalizace wt % vodivého grafitu a nanočástic feritu kobaltu v TPU matrici. Kromě toho byly vyvinuté nanokompozity použity pro aplikace stínění EMI. Dále byly zkoumány EM parametry jako komplexní permitivita a permeabilita, dielektrické ztráty, magnetické ztráty, elektrická vodivost, aby se studovala jejich závislost na EMI stínícím mechanismu vyvinutých polymerních nanokompozitů. Impedanční přizpůsobovací koeficient a konstanta útlumu pomohly předpovědět EMI stínící mechanismus připravených polymerních nanokompozitů.

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1. INTRODUCTION TO ELECTROMAGNETIC POLLUTION

During the early stages of the electronic period, there was definitely interference, such as noise, pops, or bangs on the radio. However, the degree of interference was relatively low. The proliferation of wireless communication, miniature of electronic gadgets across various sectors such as telecommunication industry, radar system, health industry has led to the emergence of the term Electromagnetic interference (EMI) or EM pollution. Additionally, after introduction of the fifth generation (5G) technology, the usage of the electronic devices have increased drastically leading to the exposure to wireless fidelity (Wi-fi) and internet and therefore enhancing the EM pollution **[1]**. Mobile phones are forbidden in the majority of hospitals and on flights owing to their interference with radiofrequency signals from other medical instruments or navigation systems **[2]**. The undesirable electromagnetic radiation emerging from the electronic equipment and their components may couple and interfere with the performance of the surrounding electronic devices. Electromagnetic interference arises when the EM signals are unintentionally transferred through radiation and/or conduction resulting in the malfunctioning of the electrically operating elements of the devices. The conduction coupling of EM signals is the primary path of interference in electric circuits and can be overcomed by designing circuits or by using frequency filters or inductors. However, EM radiations can be controlled by isolating the devices by using an EMI shielding material. Therefore, EMI shielding is focussed on attenuation of the EM waves by the EMI shielding materials by interacting with these EM waves. The influence of the EM interference ranges from disturbance of signals in broadcasting to fatal mishappenings resulting due to corrupted safety-critical control systems **[3]**. This distrupting phenomenon of EMI can led to the malfunctioning of the critical components, significant data loss, mis-interpretation of the data signals etc. Additionally, after introduction of the fifth generation (5G) technology, the usage of the electronic devices have increased drastically leading to the exposure to wireless fidelity (Wi-fi) and internet and therefore enhancing the EM pollution **[1]**. From a technological perspective, EMI is associated with issues in the industry. Aside from its impact on communication and electronic devices, EMI raises human-health risks including headache, insomnia, heart diseases, nausea etc.. Ensuring electromagnetic compatibility becomes essential because most electronic devices that are in use emit electromagnetic waves and are all susceptible to EMI issues.

To ensure the performance requirements, electromagnetic compatibility (EMC) regulations and standards have been established by the international organizations. These standards must be satisfied for commercial electronics, and one way to achieve the EMC required level is to use shielding materials. Therefore, in a global context, the interest in investments to develop advanced materials capable of, for example, reflecting or absorbing electromagnetic radiation to overcome the crescent electromagnetic pollution is evident. The impact of electromagnetic interference can be mitigating by designing an EMI shielding material which is capable of absorbing, reflecting or suppressing the radiated EM waves **[3]**. Consequently, lots of research studies are being conducted with the aim of developing multifunctional shielding materials that may present suitable mechanical properties, lower density, good processability, and, at the same time, fully satisfy aesthetics parameters.

This thesis is based on the experimental designing and development of polymer nanocomposites based on conducting and magnetic fillers for the EMI shielding applications.

2. THEORETICAL BACKGROUND OF THE STUDY

2.1 EMI shielding theory

A material's ability to attenuate EM radiation is coined as EMI shielding effectiveness. It is measured in terms of the unit decibels (dB) and is expressed as total EMI shielding effectiveness (SE_T). In general, when the incident electromagnetic waves interact with shielding material, the incident power can be divided into reflected power, absorbed power, and transmitted power. It is a ratio of logarithmic of incident power to the transmitted power as written below **[A4]**:

$$SE_T(dB) = 10\log\left(\frac{P_I}{P_T}\right) = 20\log\left(\frac{E_I}{E_T}\right) = 20\log\left(\frac{H_I}{H_T}\right) \qquad 2.1$$

where the variables P, E, and H denote the magnitudes of the power field intensity, electric field intensity, and magnetic field intensity, respectively.

According to Schelkunoff theory, the total EMI shielding effectiveness (SE_T) is contribution of three mechanism EMI shielding due to absorption SE_A, reflections SE_R and multiple internal reflections, SE_M. The total electromagnetic shielding effectiveness, SE_T of nanocomposites, is termed as their attenuation capability of the EM waves and is expressed in decibel (dB) unit as following:

$$SE_T = SE_A + SE_R + SE_M \qquad 2.2$$

Notably, if the SE_A value is higher than 10 dB or when thickness of the material is greater than the skin depth, then the contribution of multiple internal reflections is negligible.

2.1.1 Interaction of EM waves with the EMI shielding material

When an EM wave is incident on the surface of the material, there are three cases for the interaction of EM waves inside the shielding material as demonstrated in **Figure 2.1**:

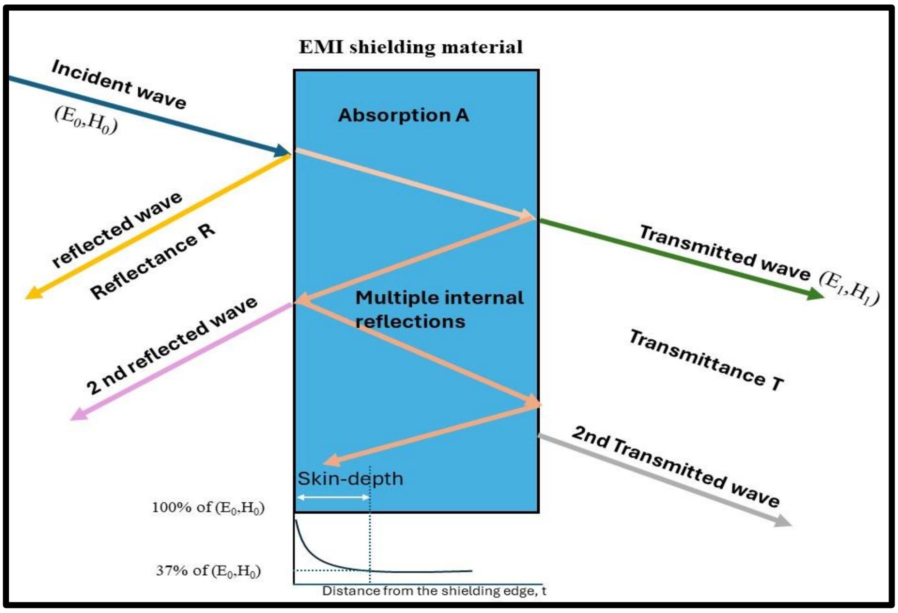

Figure 2.1 Interaction of EM waves with the EMI shielding material.

Reflection: Free charges on the surface in this mechanism are accountable for the reflection of incoming waves from the surface of the materials. When EM waves are incident on the surface of the material, then owing to the discontinuous impedance on the intersecting surface of the material with air, the incident EM waves suffers reflection. EM waves deliver energy to electrons or charged particles, inducing high-frequency oscillations in them resulting in the generation of reflecting EM waves. Therefore, reflections primarily occur at the boundary between conductive structures. Shielding due to reflection can be written as follows by simplifying the Fresnel's equation for highly conductive material**[A4]**:

$$SE_R\ (dB) = 10 \log \frac{\sigma_T}{16 f \varepsilon \mu_r} \qquad 2.3$$

where σ_T corresponds to the total electrical conductivity of the material (in S/cm) and μ_r refers to the relative magnetic permeability of the material, ε refers to the electrical permittivity of the shielding material. According to the above relation, electrical conductivity is significant for enhancing the reflection loss of the shielding material. However, reflection losses are also relying on the frequency and the permeability of the shielding material.

Multiple reflections: Some remaining part of the incident EM waves travels inside the shielding material and reaches back of the shielding surface, where it encounters the interface between the material and the air and returns back to the inner surface of the shielding material. This kind of reflection is termed as multiple reflections. Shielding due to multiple internal reflections arises due to the scattering of the radiations mostly due to inhomogeneity in the material. It can lower the EMI due to multiple reflection from front end and back end of the shielding material. It can be expressed as follows **[A4]**:

$$SE_M\ (dB) = 20\ \log_{10}(1 - e^{-\frac{2t}{\delta}}) = 20\ \log_{10}(1 - 10^{\frac{SE_A}{10}}) \qquad 2.4$$

where δ refers to the skin depth of the material.

Absorption: Some part of the incident wave doesn't get reflected and can gets absorbed inside the shielding material surface. The absorbed EM waves gets converted into dielectric loss, conduction loss and magnetic loss during the EM wave propagation. SE_A is associated with the dissipation of energies. SE_A can be expressed using following relation **[A4]**:

$$SE_A\ (dB) = 8.68 t \frac{\sqrt{f \mu_r \sigma_T}}{2} \qquad 2.5$$

where t corresponds to the thickness of the material, α corresponds to the attenuation constant and δ is skin-depth. The thickness of the nanocomposite and conductivity is responsible for the absorption and permittivity and permeability controls the absorption loss.

Skin effect

The skin effect pertains to the phenomenon where radiation that hits the surface of a conductor only affects the region close to the surface. As the radiation moves through the interior of the conductor, the electric field diminishes dramatically. Skin depth (δ) is defined as the certain distance travelled by the electromagnetic wave upto it is attenuated and drops to 1/e (37%) of its incident wave **[A4]**. It can be mathematically represented as follows **[A4]**:

$$\delta = \frac{1}{\sqrt{\pi \mu \sigma f}} \quad 2.6$$

where μ and σ refers to the magnetic permeability and electrical conductivity, respectively, of the shielding material.

2.2 Properties of EMI shielding material

2.2.1 Dielectric losses

The mechanism behind the excellent EMI shielding performance of the materials is associated with their complex permittivity and permeability. The complex permittivity constitute the real part and imaginary part of the permittivity. The real part of the permittivity (ε') refers to the electric charge storage capability of the material and is a measure of polarization centers. It is associated with the displacement current and influenced by the polarization inside the shielding material. The imaginary part of permittivity, ε" is associated with the dielectric dissipiation or losses. The increase in the conductive network with increase in the filler amount enhances the value of ε" and further improves the EMI dissipiation by absorption. The value of ε′ arises due to the different polarization including dipolar polarization, electric polarization, orientation polarization, and interfacial polarization of the material under EM waves. Electronic and ionic polarizations occurs only at very high frequency range (above 1000 GHz) **[A4]**. Owing to the existence of residual bonds and defects created during chemical synthesis, electrons are not homogeneous inside the material resulting in orientation polarization. Interfacial polarization originates from the existence of the functional groups, defects, and numerous interfaces presents owing to the dissimilar dielectric constant at the interface on the surface of the fillers in polymer nanocomposites. The dielectric permittivity can increase the dielectric losses due to the mismatch in the electrical conductivity of the non-conductive polymer and conductive filler **[A4]**. This dissimilarity between the electrical conductivity of filler and polymer matrix induces polarization and accumulation of charges at their interface. Dielectric losses, $tan\delta_{\varepsilon}$ originates from the polarization and associated relaxation phenomena occuring in the developed nanocomposites. The dielectric losses can be evaluated using the following expression**[A4]**:

$$tan\delta_{\varepsilon} = \varepsilon''/\varepsilon' \qquad 2.7$$

Further, Debye theory is used to analyze the role of relaxation mechanisms inside the polymer nanocomposites for EMI shielding. According to Debye theory, the value of real and imaginary permittivity can be expressed as follows:

$$\varepsilon' = \varepsilon_{\infty} + \frac{\varepsilon_s - \varepsilon_{\infty}}{1 + (\omega\tau)^2} \qquad 2.8$$

$$\varepsilon' = \varepsilon_{\infty} + \frac{\varepsilon_s - \varepsilon_{\infty}}{1 + (\omega\tau)^2} \qquad 2.9$$

$$\varepsilon'' = \frac{\varepsilon_s - \varepsilon_{\infty}}{1 + (\omega\tau)^2}\omega\tau + \frac{\sigma}{\omega\varepsilon_o} \qquad 2.10$$

where ε_{∞} is the relative dielectric permittivity at an infinite frequency, ε_s is the static dielectric permittivity, ω represents angular frequency, σ refers to the electrical conductivity and τ stands for the polarization relaxation time, respectively. On neglecting the role of σ to ε" and elimination of ωτ, the equation between ε' and ε" can be rewritten as follows **[A4]**:

$$(\varepsilon' - \frac{\varepsilon_s + \varepsilon_{\infty}}{2})^2 + (\varepsilon'')^2 = (\frac{\varepsilon_s - \varepsilon_{\infty}}{2})^2 \qquad 2.11$$

According to above expression, the plot of ε' versus ε" called as Cole-Cole plot and should be a semicircle. Each semicircle represents Debye relaxation phenomenon and can be enhanced with interfaces inside the polymer nanocomposites, which further helps in improving the EM wave absorption. Distorted semi-circles are due to the heterogeneous surface polarization and motion of conducting electrons that improves permittivity of the material.

2.2.1.1 Electrical conductivity

On the basis of free-electron theory, the dielectric losses are prominently generated from conduction losses according to the following expression **[A4]**:

$$\varepsilon'' = \sigma/2\pi\varepsilon_0 f \qquad 2.12$$

The above equation demonstrates that higher value of electrical conductivity improves the value of imaginary permittivity which represents the dielectric dissipiation of EM waves.

2.2.2 Magnetic losses

The real part of the permeability μ' is responsible for the magnetic energy storage capacity of the nanocomposites. The imaginary part of the permeability μ" signifies the dissipation ability of magnetic energy. The magnetic losses tangent can be evaluated using the following expression**[A4]**:

$$tan\delta_\mu = \mu''/\mu' \qquad 2.13$$

The magnetic loss is generally controlled by hysteresis loss, domain wall loss, eddy current, natural resonance, exchange resonance, and anisotropic energy, residual loss in the nanocomposites. The hysteresis loss arises from the time lag of the magnetization vector M behind the magnetic field vector H. However, domain-wall resonance arises at lower GHz frequencies. Spin resonance exists at higher frequencies. In X-band region, the magnetic loss can be attributed to the eddy current loss, natural resonance, exchange resonance at higher frequencies.

2.2.3 Eddy current losses

The eddy current loss originates due to the current induced by the magnetic part of the incident EM field, and can be assessed using the following expression**[A4]**:

$$C_o = \mu''(\mu')^{-2} f^{-1} \qquad 2.14$$

According to the above equation, if the magnetic loss is originating only due to eddy current, then the plot of C_o remains constant with a change in frequency.

2.2.4 Natural resonance

Natural resonance is responsible for the magnetic losses, which can be evaluated as follows**[A4]**:

$$2\pi f_r = \gamma H_a \qquad 2.15$$

where γ denotes gyromagnetic ratio and H_a represents anistropy energy. Further, anistropy energy has strong dependence on the saturation magnetization according to the following expression:

$$H_a = 4|K_1|/3\mu_o M_s \qquad 2.16$$

The inversely proportional relationship between H_a and M_s indicates enhancement of anisotropy energy with decrease in saturation magnetization. Further, the anisotropy energy of the nanosized materials would be enhanced owing to its surface anisotropy field effect. Therefore, anisotropy energy is dependent on particle size as well as saturation magnetization of the material. Moreover, natural resonance is observed at low frequency range (2-10 GHz).

2.2.5 Exchange resonance

According to the theory of Aharoni, the exchange resonance can be given by the following expression**[A4]**:

$$f_{exc} = (C.\frac{u_{kn}^2}{R^2 M_s} + H_0 - aM_s + \gamma H_a)^{\gamma}/_{2a} \qquad 2.17$$

where f_{exc} refers to the exchange frequency; C is exchange constant; u_{kn} is the roots of the derivatives of the spherical basel function; R is the radius of the particle absorber; H_0 is magnetocrystalline anisotropy field, and H_a represents anistropy energy.

At higher frequencies (over 10 GHz), exchange resonance is observed. Notably, when particle size decreases, exchange resonance shifts towards higher frequencies. Thus, in general, the exchange resonance is correlated with the oscillations observed at higher frequencies.

2.2.6 Attenuation constant

Apart from high dielectric and magnetic losses, attenuation constant (α) is a critical factor to be fulfilled by the EMI shielding material for achieving an outstanding EMI absorption. In general, the attenuation constant (α) can provide the electromagnetic wave attenuation capacity of the shielding material when an EM wave is incident on the surface of shielding material. The high value of α corresponds to the high microwave dissipiation through strong absorption. It can be calculated by the following expression **[A4]**:

$$\alpha = \frac{\sqrt{2}\pi f}{c}\sqrt{(\mu''\varepsilon'' - \mu'\varepsilon') + \sqrt{(\mu''\varepsilon'' - \mu'\varepsilon')^2 + (\varepsilon'\mu'' + \varepsilon''\mu')^2}} \tag{2.18}$$

where c stands for speed for light in vacuum. According to above expression, high value of μ'' and ε'' is significant to attain high attenuation of EM wave. Therefore, its very important to tune the EM parameters for improving the EMI absorption properties.

2.2.7 Impedance matching

The impedance of an electromagnetic wave is determined by the ratio of the electric and magnetic field's transverse components. The typical impedance of free space is approximately 377 Ω **[4]**. Well-matched impedance should be attained between permittivity and permeability of the shielding material to achieve good EMI shielding characteristics. A good impedance matching is a mark of good EM wave absorption. Enhancing impedance matching between free space and the shield allows for greater penetration of electromagnetic waves into the shield, resulting in the dissipation of energy as heat through internal attenuation **[4]**. For ensuring the efficient EM waves entered to the EMI shielding material so that EM waves can easily enter the surface of the material rather than reflecting, the value of impedance matching should be 1 or close to 1. The value of impedance matching coefficient controls how much EM wave can propagate inside the EMI shielding material and is given by the following expression **[A4]**:

$$Z = \sqrt{\mu_r/\varepsilon_r} = \sqrt{\sqrt{(\mu'^2 + \mu''^2)}/\sqrt{(\varepsilon'^2 + \varepsilon''^2)}} \tag{2.19}$$

2.2.8 Shielding efficiency

As mentioned before, a minimum of 20 dB of total EMI SE is required for commercial applications such as in aircraft, satellite, telecommunications, and defense industry. In light of this, the evaluation of the shielding efficiency can be performed using the following expression **[5]**:

$$Shielding\ Efficiency\ (\%) = 100 - \left(\frac{1}{10^{\frac{SE}{10}}}\right) \times 100 \tag{2.20}$$

An EMI shielding material with 20 dB of EMI SE is known to be able to block 99% of incident EM waves while transmitting less than or equal to 1 percent of EM waves.

2.2.9 Thickness and Density

Density and weight are the two crucial factors that influence the performance of the EMI shielding material and play a significant role in aerospace and electronics applications. For a better assessment of the EMI shielding performance of the material, the specific shielding effectiveness (SSE) and absolute shielding effectiveness (SSE_t) were also calculated using the following equations **[A1,A4]**:

$$\mathrm{SSE} = \mathrm{SE} \times \mathrm{V/m} \tag{2.21}$$

$$\frac{SSE}{t} = SE \times \frac{V}{m \times t} \tag{2.22}$$

where m symbolizes mass, v refers to volume and t is the thickness of the material.

2.3 Test method for EMI shielding

Shielding effectiveness for any composite shielding material is mostly widely measured by waveguide transmission method. In this method, two rectangular waveguide adapters are connected to a network analyzer via a coaxial cable. The rectangular shaped sample is placed inside the waveguide of a network analyzer and the signals are transmitted through the waveguide port 1. Further, the reflected signals from the sample in the conductive mold are received at port 2. The advantage of utilizing vector network analyzer over scalar network analyzer is that it can be utilized to evaluate the complex permittivity and permeability of the material. Notably, sample holders of different sizes are utilized for different frequency ranges. As mentioned, the device features two ports, and it allows for the measurement of four parameters: S_{11} (forward reflection parameter), S_{22} (backward reflection parameter), S_{12} (forward transmission parameter), and S_{21} (backward transmission parameter). These parameters are represented as complex numbers. The evaluation of reflection coefficient R, transmission coefficient T and absorption coefficient A can be performed using the following equation **[A4]**:

$$R = |S_{11}|^2 = |S_{22}|^2 \quad 2.23$$

$$T = |S_{12}|^2 = |S_{21}|^2 \quad 2.24$$

$$A = 1 - R - T \quad 2.25$$

The calculations of shielding effectiveness of the composites are dependent on the scattering parameters (S) obtained experimentally. Utilizing the scattering parameters, the value of SE_T , SE_A and SE_R are evaluated using the following expressions:

$$SE_T = 10\log_{10}\left(\frac{1}{|S_{12}|^2}\right) = 10\log_{10}\left(\frac{1}{|S_{21}|^2}\right) = -10\log_{10} T \quad 2.26$$

$$SE_R = 10\log_{10}\left(\frac{1}{|S_{11}|^2}\right) = -10\log_{10}(1-R) \quad 2.27$$

$$\mathrm{SE_A} = 10\log_{10}\left(\frac{1-|S_{11}|^2}{|S_{12}|^2}\right) = -10\log_{10}\left(\frac{T}{1-R}\right) \quad 2.28$$

2.4 Construction strategies for EMI shielding material

2.4.1 Polymer nanocomposites

Polymeric materials provide the benefits of being lightweight, resistant to corrosion, and easily processed. Polymers of various kinds, such as plastics, rubbers, and fibers, are commonly employed as matrices for producing polymer nanocomposites. Pure polymers may not be practical for use. Polymer nanocomposites (PNCs) are highly in demand by the industries and researchers for their valuable contribution to packaging, transportation, sensors, catalysis, defense systems, aerospace, and electromagnetic interference (EMI) shielding. PNCs are hybrid materials with the dispersion of nanofillers inside the polymer matrix. The filler can be reinforced into the polymer in form of particles, continuous layered materials, and filaments. These nanofillers possessing at least one dimension in the nanometer range, are utilized as a supplement in the polymer matrix. High flexibility, chemical and corrosion resistance, light weight, and elasticity of thermoplastic polyurethane (TPU) makes it as an ideal choice for industrial and biomedical applications **[6]**. Therefore, employing a composite material consisting of a polymeric matrix and an embedded filler as demonstrated in **Figure 2.2** could be an effective approach for EMI shielding applications. To enhance conductivity of the polymers, conductive additives like graphene are commonly used. The significance of utilizing conductive fillers in a polymeric matrix is prominent, as the attainment of high electrical conductivity plays a pivotal role in enhancing the performa`nce of polymer composites in electromagnetic interference (EMI) shielding applications **[7]**.

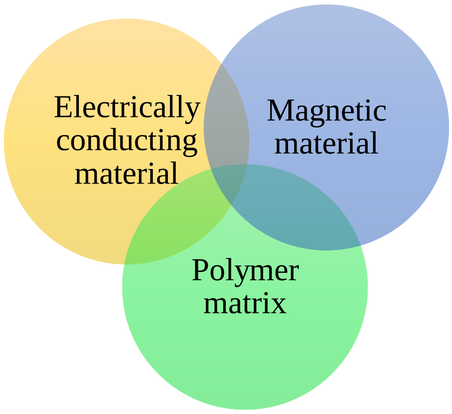

Figure 2.2 Construction strategie of EMI shielding material with efficient EM wave absorption.

Moreover, introduction of magnetic filler enhances the magnetic losses inside the polymer nanocomposite system. Incorporating dielectric and magnetic material as the filler will enhance the microwave absorption capability of the polymer matrix through their synergestic effect. The multi-component composite, comprising a polymer together with conducting and magnetic filler, effectively mitigates electromagnetic interference along with green EMI shielding based on absorption **[8]**.

2.5 Novel magnetic filler

2.5.1 Spinel ferrites

Within the category of magnetic materials that exhibit extensive technological applications, ferrites garner considerable interest and are irreplaceable due to their low cost, stability, and widespread utilization in high-frequency applications. Spinel ferrites are considered a significant inorganic nanomaterial due to its remarkable magnetic, electronic, electrical, catalytic, and optical capabilities. Spinel ferrites have been recognized as a potential candidate to be utilized as a magnetic filler due to their abundant availability, low cost, excellent dispersibility, environmentally friendly nature, non-toxicity, and impressive electromagnetic characteristics **[9]**. The superparamagnetic characteristics of ferrite nanoparticles reveals remanence and coercivity; thus, the developed polymer nanocomposites exhibit exceptional performance. Spinel ferrites are receiving significant research interest due to their remarkable chemical stability and magnetic and electrical properties. These properties include high saturation magnetization, high squareness ratio, large magneto-crystalline anisotropy, low coercivity, high electric resistivity, low eddy current losses, elevated curie temperature, and mechanical hardness. Spinel ferrites are commonly employed in microwave absorption due to their substantial magnetic losses and notable resistance. The morphology, dimensions, surface characteristics, and aspect ratio of nanoparticles play a crucial role in modulating these interactions and influencing the features of the resulting systems. The functionalization of nanofillers is employed to modify interfacial states and enhance the mechanical and physical properties of polymer nanocomposites, owing to the significant interfacial area between polymers and nanofillers. Several approaches have been utilized to alter spinel ferrite based nanocomposites such as manipulating the composition by incorporating a guest metal cation, adopting different synthesis routes, and alterning the surface features of the structure. The magnetic properties of ferrites are influenced by the composition and quantity of guest metal cations, as well as their arrangement within the tetrahedral and octahedral sites. Further, several factors influences the magnetic properties of the ferrites including method of synthesis, grain size, magnetic nature of dopant, existence of secondary phases and inturn can influence the EMI shielding characteristics of the developed polymer nanocomposites **[10]**.

2.6 Novel conducting fillers

2.6.1 Graphite, GO and rGO

Graphite ore exists in a crystalline structure and is a naturally occurring allotrope of carbon. The material exhibits a layered composition, with carbon atoms grouped in a hexagonal configuration within each layer. The layers are organized in an AB sequence **[11]**. The layers are interconnected through van der Waals interactions, which are generated by delocalized π-orbitals and therefore enables the layers to be readily separated. Graphite is a highly versatile material that is used in a wide range of energy applications, including batteries, fuel cells, water purification, fiber optics, and supercapacitors **[12,13]**. It possesses exceptional thermal and electrical conductivity, as well as remarkable strength and stiffness **[14]**. Graphite, due to its exceptional electrical conductivity, mechanical strength, high specific surface area, high carrier mobility, and thermal properties, can be used as a filler in polymer nanocomposites to enhance their dielectric loss and provide a conductive pathway.

Graphene, being a remarkable class of conductive fillers, garners considerable interest due to its exceptional structure and properties. The graphene sheet is a hexagonal two-dimensional carbon nanofiller composed of a planar honeycomb lattice and a one-atom-thick sheet of sp^2-bonded carbon atoms**[11]**. Graphene possesses exceptional characteristics compared to traditional nanofillers, including higher thermal conductivity and Young's modulus, as well as superior mechanical and electronic properties, significant specific surface area and aspect ratio, EMI shielding capability, low coefficient of thermal expansion, and flexibility **[15]**. Graphene oxide (GO) is a carbon structure consisting of layers with oxygen-containing functional groups (=O, -OH, -O-, -COOH) bonded to both sides of the layer and the edges of the plane. Graphene oxide (GO), like other 2D carbon materials, can exist in either a single layer or multilayer structure. GO is recognized for its composition of both sp^2 and sp^3 hybridized carbon atoms. Moreover, GO has an amorphous structure, indicating that the oxygen functional groups are randomly bonded to the graphene plane **[16]**. When the sp^3 hybridization of carbon atoms in GO/graphite changes into the sp^2 in a single or few layers of graphene, it results in an increase in free electrons within the structure, hence enhancing the inherent electrical conductivity. The synthesis procedure of graphene contributes to a consistent enhancement in electrical conductivity when transitioning from graphite to graphene. GO exhibits both amorphous and crystalline defect areas, which reveal sp^3-hybridized carbon and oxygen functional groups **[17]**.

At a high degree of oxidation, the structure and chemical constituents of GO are unstable. Reducing GO results in significant alterations to the chemical composition of the sheet, hence creating possibilities for customizing its characteristics **[18]**. Reduction treatment minimizes the oxygen concentration, surface charge, and hydrophilicity of GO, resulting in the production of rGO with improved optical absorbance and restored electrical conductivity.

3. AIM OF THE DOCTORAL THESIS

The thesis aims to investigate the structural, morphological, and electromagnetic properties of advanced polymer nanocomposites consisting of spinel ferrite nanoparticles and graphite or reduced graphene oxide for electromagnetic interference (EMI) shielding application. The stepwise tasks to achieve the objectives of the doctoral thesis are as follows:

i. Synthesis of various spinel ferrite nanoparticles with incorporation of various dopants ions such as Zn^{2+} and Cu^{2+} in different stoichiometry, using sonochemical approach under various synthesis conditions. Characterization of synthesized spinel ferrite nanoparticles using XRD technique, Raman spectroscopy, FTIR spectroscopy, X-ray photoelectron spectroscopy, Transmission electron microscopy (TEM) and Vibrating sample magnetometer (VSM), etc.

ii. Synthesis of electrically conductive filler reduced graphene oxide, and its structural, morphological investigation by XRD, XPS, and SEM characterization techniques.

iii. Developing polymer nanocomposites consisting of synthesized spinel ferrite nanoparticles and graphite or reduced graphene oxide using a melt-mixing approach and further its characterizations such as XRD technique, Raman spectroscopy, field emission-scanning electron microscopy (FE-SEM), vibrating sample magnetometer (VSM), etc.

iv. Investigating the scattering parameters (S_{11}, S_{12}, S_{21}, S_{22}), complex permittivity (ε_r), and complex permeability (μ_r), with a PNA-L network analyzer (Agilent N5230A) using an X-band (WR 90) waveguide within the frequency range 8.2-12.4 GHz. Evaluating the electromagnetic interference (EMI) shielding effectiveness (SE_T) by the recorded scattering parameters (S_{11}, S_{12}, S_{21}, S_{22}) in the X-band frequency range. Assessing the complex permittivity(ε_r), and permeability (μ_r) by using the Nicolson-Ross-Weir technique.

v. Investigation of the relation among complex permittivity(ε_r), complex permeability (μ_r), electrical conductivity($\sigma_{a.c.}$), dielectric loss (tanδ_ε), magnetic loss (tanδ_μ), eddy current loss (C_0), skin-depth (δ), attenuation constant (α) and impedance matching coefficient (Z),with EMI shielding effectiveness (SE_T) of developed nanocomposites.

4. EXPERIMENTAL METHODS

4.1 Preparation of Zn^{2+}-Substituted $CoFe_2O_4$ Nanoparticles based TPU nanocomposites along with rGO

4.1.1 Synthesis of Zn^{2+}- substituted $CoFe_2O_4$ nanoparticles

Zinc substituted $CoFe_2O_4$ nanoparticles ($CoFe_2O_4$, $Co_{0.67}Zn_{0.33}Fe_2O_4$, and $Co_{0.33}Zn_{0.67}Fe_2O_4$) were prepared by sonochemical synthesis route as demonstrated in **Figure 4.1**. The stoichiometric ratio of the precursor cobalt nitrate and the dopant precursor zinc nitrate (sourced from Alfa Aesar GmbH and Co KG, Germany), was dissolved in deionized water through stirring. A solution of NaOH was prepared and was allowed to react with the dissolved reaction mixture under the influence of sonication waves for a duration of 1 hour. Further, the excess of the formed residue was removed by washing with DI water and ethanol. The excess of precipitate was eliminated by centrifugation. This process was repeated several times. Then, the obtained product was allowed to dry in an oven at 60 °C for 24 h. The synthesized nanoparticles consisting different compositions such as $CoFe_2O_4$, $Co_{0.67}Zn_{0.33}Fe_2O_4$, and $Co_{0.33}Zn_{0.67}Fe_2O_4$ were designated as CZ1, CZ2, and CZ3 nanoparticles, respectively **[A1]**.

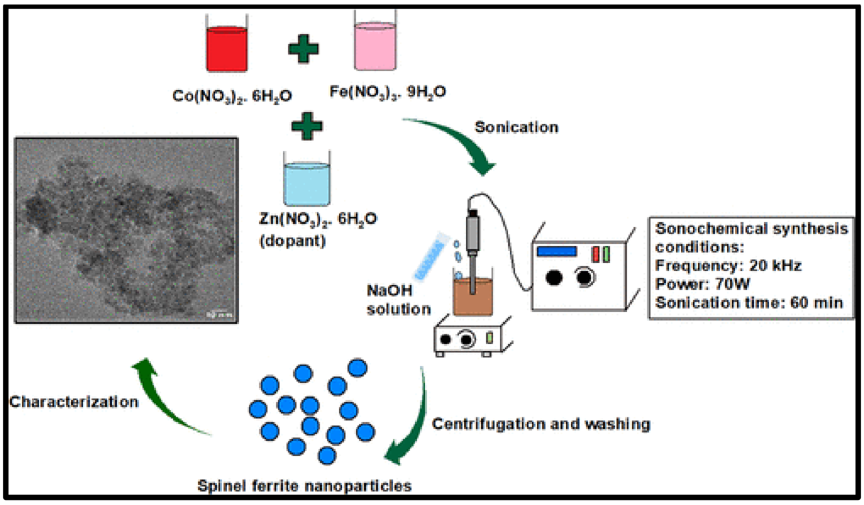

Figure 4.1 Synthesis of Zn^{2+}- Substituted $CoFe_2O_4$ ferrite nanoparticles ***[A1]****.*

4.1.2 Synthesis of reduced graphene oxide (rGO)

Graphene oxide was synthesized using Hummer's technique by commerically available expandable graphite flakes (procured from Sigma-Aldrich, Germany). Graphite flakes (3 g) and (1.5 g) sodium nitrate (obtained from Lach-Ner, Czech Republic) were disseminated in concentrated sulfuric acid (75 mL). Following that, 9 g of potassium permanganate was added slowly to the above-prepared solution and left at room temperature for 12 hours. H_2O_2 (30%, 2 mL) was utilized to further treat the above-mentioned mixed solution. To remove the remaining ions, the resulting mixture was rinsed with deionized water. The final product was annealed for 24 hours at 60 °C. Further, for reduction, 3 g of obtained graphene oxide (GO) was mixed in 200 mL of deionized water by stirring. 10 g of vitamin C (purchased from Dr. Kleine Pharma GmbH, Germany), was added to the reaction mixture and further the solution was kept for stirring for 3 hours at a constant temperature of 100 °C. The reaction mixture was allowed to cool down and further it was washed using ethanol and deionized water. Following that, the solution was centrifuged for 20 minutes at 8000 rpm. The product was subsequently dried for 14 hours at 60 °C in a vacuum oven **[A1]**.

4.1.3 Preparation of CZ1-rGO-TPU, CZ2-rGO-TPU and CZ3-rGO-TPU polymer nanocomposites

The polymer nanocomposites were developed via melt-mixing technique (**Figure 4.2**) in which thermoplastic polyrethane (TPU) (Elastollan C80A10) was used as polymer matrix. 20wt% of the fillers composing spinel ferrites and rGO in a 9:1 wt% ratio, was utilized to develop the polymer nanocomposites. A microcompounder possessing twin screws was employed to prepare the polymer nanocomposites. In the first phase of the process, both the fillers and the TPU polymer was dried at 90 °C for 12 hours in a vaccum oven. The spinel ferrite nanoparticles and reduced graphene oxide was transferred into the hopper simultaneously with TPU. The melt mixing of the fillers with the TPU polymer was programmed to be perform at a speed of 150 rpm and a temperature of 200 °C for 7 min. A set of three polymer nanocomposite was prepared and were designated as CZ1-rGO-TPU, CZ2-rGO-TPU, and CZ3-rGO-TPU. In last, the desired dimensions (22.9 mm × 10.2 mm × 0.8 mm) of the polymer nanocomposites were obtained by hot-compression moulding. **Figure 4.3** demonstrates the digital picture of the developed TPU-based nanocomposites with spinel ferrite nanoparticles with

Reduced graphene oxide showing the dimension, lightweight, and flexibility **[A1]**.

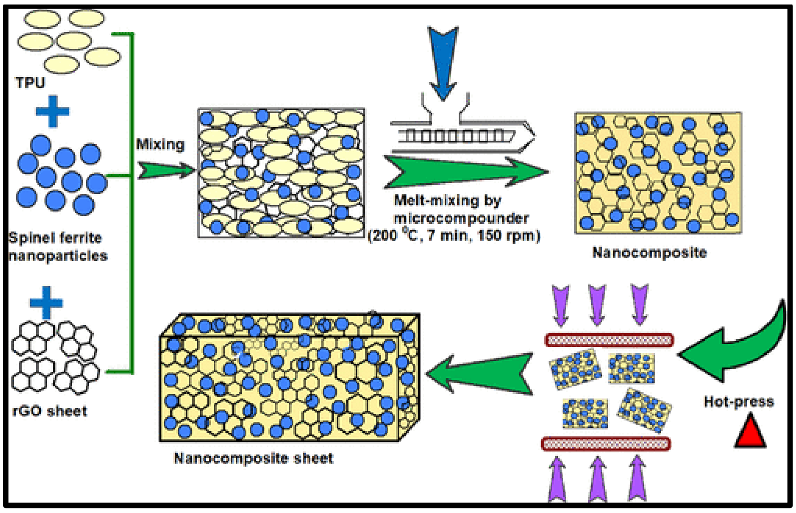

*Figure 4.2 Schematic illustration of the preparation of TPU-based nanocomposites with spinel ferrite nanoparticles and Reduced graphene oxide **[A1]**.*

*Figure 4.3 Pictorial representation of the developed TPU-based nanocomposite with spinel ferrite nanoparticles and reduced graphene oxide demonstrating the dimension, lightweight, and flexibility **[A1]**.*

4.2 Preparation of $Cu_xCo_{1-x}Fe_2O_4$ (x = 0.33, 0.67, 1) Spinel Ferrite Nanoparticles based Thermoplastic Polyurethane Nanocomposites with Reduced Graphene Oxide

4.2.1 Synthesis of $Cu_xCo_{1-x}Fe_2O_4$ (x = 0.33. 0.67, 1) spinel ferrite nanoparticles

The sonochemical approach was used to synthesize $Cu_{0.33}Co_{0.67}Fe_2O_4$, $Cu_{0.67}Co_{0.33}Fe_2O_4$, and $CuFe_2O_4$ nanoparticles **[A2]**. Spinel ferrite nanoparticle formation, appropriate proportion of the precursors cobalt nitrate, copper nitrate, and iron nitrate (procured from Alfa Aesar GmbH &

Co. KG (Karlsruhe, Germany)) were mixed with 60 mL of deionized water and stirred for at room temperature to obtain a clear solution. A NaOH solution was prepared and gently added to the mixture, accompanied by stirring for another few minutes. With the addition of a base solution, the solution thickened into a precipitate. Further, the reaction mixture was subjected to ultrasonic irradiation at 20 kHz and 70W power for 1 hour. The precipitate was then cooled, rinsed with deionized water, and centrifuged at 7000 rpm for 15 minutes. To remove any leftover contaminants, this process was done numerous times and further the obtained product was dried at 60 °C in an oven for 24 h. The synthesized $Cu_xCo_{1-x}Fe_2O_4$ (x = 0.33, 0.67, 1) spinel ferrite nanoparticles with varied compositions, such as $Cu_{0.33}Co_{0.67}Fe_2O_4$, $Cu_{0.67}Co_{0.33}Fe_2O_4$, and $CuFe_2O_4$, were named as CuCoF1, CuCoF2, and CuF3 **[A2]**.

4.2.2 Synthesis of reduced graphene oxide (rGO)

rGO was synthesized using chemical reduction of graphene oxide (GO) by Vitamin C as described in the **section 4.1.2**.

4.2.3 Preparation of CuCoF1-rGO-TPU, CuCoF2-rGO-TPU, and CuF3-rGO-TPU nanocomposites

The TPU (Elastollan C80A10) based polymer nanocomposites were prepared by melt-mixing technique in which the 20-wt% of the fillers was maintained out of which spinel ferrites nanoparticles and rGO was in 9:1 wt% ratio. Melt-compounding was performed using a twin-screw extruder with a capacity of 5 cm^3 at 150 rpm of screw speed and a temperature of 200 °C as demonstrated in **Figure 4.4**. Both the fillers and the TPU was dried at 90 °C for 12 h in a vacuum oven before initiating the melt-mixing process. The pre-dried fillers and the TPU were fed to the hopper and melt-mixed for 7 minutes. A set of three nanocomposite, designated as CuCoF1-rGO-TPU, CuCoF2-rGO-TPU, and CuF3-rGO-TPU, were prepared **[A2]**. Further, the prepared polymer nanocomposites were shaped into rectangular form (22.9 mm × 10.2 mm × 1.0 mm) using hot-compression moulding.

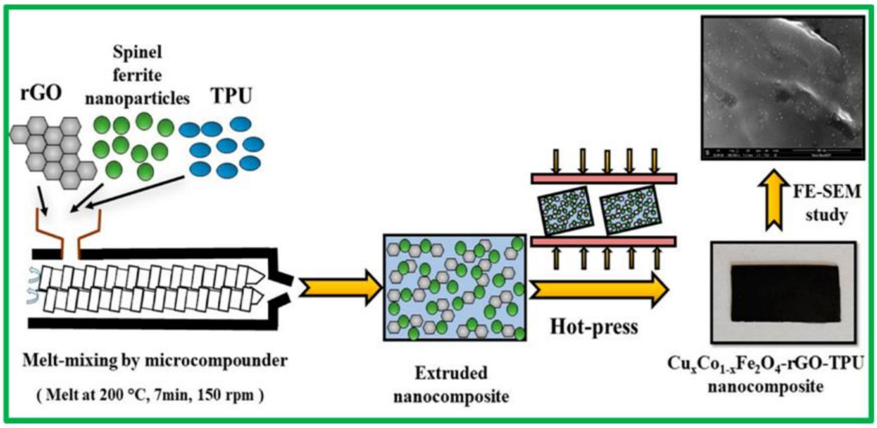

Figure 4.4 Schematic representation of the preparation of $Cu_xCo_{1-x}Fe_2O_4$ - rGO-TPU nanocomposite ***[A2]****.*

4.3 Preparation of $CoFe_2O_4$ Nanoparticles along with graphite in TPU matrix

4.3.1 Synthesis of $CoFe_2O_4$ nanoparticles

Spinel ferrite nanoparticles were synthesized using the sonochemical technique. In a typical procedure, appropriate stoichiometric amounts of precursors such as cobalt nitrate and iron nitrate were mixed with 120 mL of deionized water. An aqueous solution of NaOH was prepared and added to the above mixture slowly, accompanied by stirring for a few seconds. Further, it was exposed to ultrasonic irradiation (ultrasonic homogenizer UZ SONOPULS HD 2070) for 60 minutes. Afterwards, the precipitate was allowed to cool down and further centrifuged. This process was repeated several times to remove any remaining impurities. The spinel ferrite nanoparticles were collected by further drying the acquired product.

4.3.2 Preparation of graphite-ferrite (GF)-TPU nanocomposites

In this procedure, GF-TPU nanocomposites were developed using the melt-mixing technique (**Figure 4.5**). A polyether based TPU (Elastollan 1195 A) was employed as a polymer matrix for developing nanocomposites. To prepare the TPU-based nanocomposites, the content of the fillers was maintained at 50wt.%, which included the varying content wt.% of $CoFe_2O_4$ nanoparticles and graphite in the TPU matrix. Four sets of nanocomposites were prepared with varying content wt.% of: 5% $CoFe_2O_4$ nanoparticles + 45% graphite, 10% $CoFe_2O_4$ nanoparticles + 40% graphite, 15% $CoFe_2O_4$ nanoparticles + 35% graphite, and 20% $CoFe_2O_4$ nanoparticles + 30% graphite, which were designated as sample GF5-TPU, GF10-TPU, GF15-TPU, and GF20-TPU, respectively. Further, the developed TPU-based nanocomposites were obtained in rectangular-shaped sheets using the compression-molding technique.

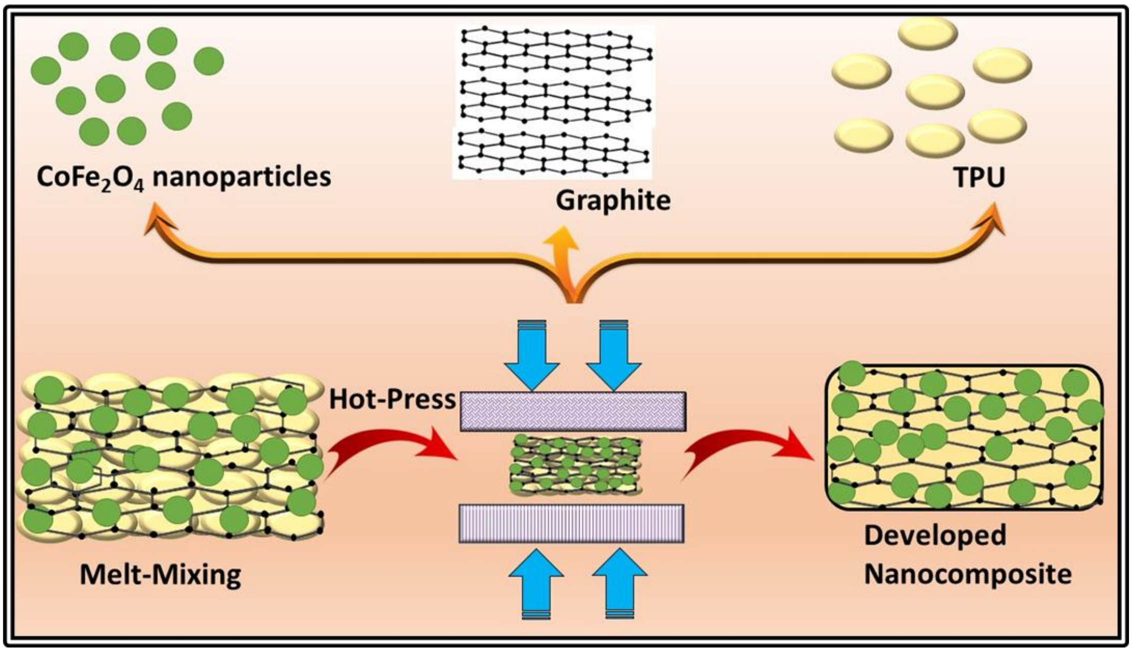

*Figure 4.5 Schematic representation of the development of the GF-TPU nanocomposites **[A3]**.*

4.4 General characterization techniques

The spinel ferrite nanoparticles and the conducting filler such as graphene oxide or reduced graphene oxide and further their developed polymer nanocomposites was studied comprehensively using the following instrumentation techniques:

1. Structural study:
   i. X-ray Diffraction technique (XRD),
   ii. Raman Spectroscopy,
   iii. FTIR spectroscopy,
   iv. X-ray Photoelectron spectroscopy (XPS).

2. Morphology and elemental study:
   i. Scanning Electron microscopy (SEM),
   ii. Transmission electron microscopy (TEM),
   iii. Energy dispersive x-ray spectroscopy (EDX),

iv. Elemental mapping.

3. The magnetic characteristics analysis: vibrating sample magnetometer (VSM) at room temperature in an applied magnetic field up to 10 kOe.

4. A PNA-L network analyzer (Agilent N5230A) over a frequency range of 8.2–12.4 GHz was utilized to measure scattering parameters. The recorded scattering parameters were utilized for calculation of EMI shielding effectiveness (SE_T, SE_A, SE_R). Power coefficient R, T, A were evaluated. In addition, shielding efficiency (%), specific and absolute EMI shielding effectiveness were calculated.
5. Complex permittivity and permeability was evaluated using the Nicolson-Ross-Weir technique. Electrical conductivity, cole-cole plot, dielectric loss, magnetic loss, skin depth, attenuation constant, and impedance matching coefficient were evaluated.
6. Thermal stability test: Thermal Thermogravimetry.
7. Mechanical characteristics: Testometric universal-testing machine.

5. DISCUSSION

5.1 Thermoplastic Polyurethane Nanocomposites with Zn^{2+} - Substituted $CoFe_2O_4$ Nanoparticles and Reduced Graphene Oxide as Shielding Materials against Electromagnetic Pollution

The objectives of this work were designing of a high-performance TPU based nanocomposites with cobalt ferrite nanoparticles as primary filler and reduced graphene oxide as a secondary filler. This work is focussed on a detailed investigation on the impact on the EMI SE performance of TPU nanocomposites with rGO and Zn^{2+}-substituted $CoFe_2O_4$ magnetic spinel ferrite nanoparticles. EMI shielding materials comprising of both the dielectric and magnetic materials will enhance the EMI shielding performance and improve impedance matching by effectively leveraging the synergies and complementarities between dielectric and magnetic loss. $CoFe_2O_4$ exhibits high saturation magnetization and high magnetocrystalline anisotropy which results in its high value of complex permeability at higher GHz frequency range. To tune the magnetic and dielectric properties of the $CoFe_2O_4$ nanoparticles, Zn^{2+} ions were incorporated in the $CoFe_2O_4$ structure. The incorporation of diamagnetic Zn^{2+} ions in the $CoFe_2O_4$ structure can lead to changes in the structural and electromagnetic property related to the distribution of cations at octahedral and tetrahedral sites. The advanced TPU-based nanocomposites were developed for EMI shielding application by employing Zn^{2+}-substituted $CoFe_2O_4$ spinel ferrite nanoparticles (CZ1, CZ2, and CZ3) and rGO as nanofillers. These nanocomposites are lightweight, flexible, and exhibit excellent performance. The nanocomposites with a thickness of 0.8 mm achieved maximum total shielding effectiveness (SE_T) values of 48.3 dB, 61.8 dB, and 67.8 dB for CZ1-rGO-TPU, CZ2-rGO-TPU, and CZ3-rGO-TPU, respectively, in the frequency range of 8.2–12.4 GHz. The CZ3-rGO-TPU nanocomposite demonstrated a specific shielding efficiency (SSE) value of 58 dB cm^3/g on evaluation. The absolute shielding efficiency (SSE_t) of CZ3-rGO-TPU was determined to be 730 dB cm^2/g. The CZ3-rGO-TPU nanocomposite exhibited an EMI shielding efficiency of 99.9999%. The dependence of complex permittivity, complex permeability, dielectric losses, magnetic losses, eddy current losses to the EMI shielding properties of the developed nanocomposites was also comprehensively investigated. The high electrical conductivity and large surface area of rGO can significantly contribute in improving the EMI shielding properties of the developed nanocomposite. The generated defects polarization relaxation and electron dipole relaxation can facilitate the EM wave absorption. Owing to the flexibility, stretchability, and good mechanical characteristics of the TPU polymer, contributed in attaining the flexibility in the developed nanocomposite. The CZ3-rGO-TPU nanocomposite achieves enhanced impedance matching by striking a superior balance between dielectric and magnetic loss, hence leading to excellent EMI shielding capabilities. The meticulously engineered nanocomposite, which is both lightweight and flexible, is highly suitable for practical application in several domains such as aviation, satellite, telecommunications, and radar stealth technology **[A1]**.

5.2 $Cu_xCo_{1-x}Fe_2O_4$ (x = 0.33, 0.67, 1) Spinel Ferrite Nanoparticles Based Thermoplastic Polyurethane Nanocomposites with Reduced Graphene Oxide for Highly Efficient Electromagnetic Interference Shielding

Polymer nanocomposites, in combination with magnetic and dielectric nanofillers, have emerged as a promising avenue in various fields. In this study, hybrid filler systems consisting of $Cu_xCo_{1-x}Fe_2O_4$ (where x = 0.33, 0.67, 1) spinel ferrite nanoparticles and reduced graphene oxide (rGO) within a thermoplastic polyurethane (TPU) matrix was employed. It is worth noting that the magnetic moment and ionic radius of Cu^{2+} ions differ from those of Co^{2+} ions **[19]**. Anticipated improvements in the magnetic and structural properties of mixed ferrites were expected with the substitution of Cu^{2+} in $CoFe_2O_4$, due to the contrasting magnetic properties and ionic radius of Cu^{2+} ($3d^9$) and Co^{2+} ($3d^7$) **[19]**. To facilitate the fabrication of thermoplastic polyurethane nanocomposites, the primary plan was to utilize three distinct samples of spinel ferrite nanoparticles. These samples comprised of one with a minimal concentration of Cu^{2+}in $CoFe_2O_4$ nanoparticles, another with an increased amount of Cu^{2+} in $CoFe_2O_4$ nanoparticles, and a final sample composed solely of pure $CuFe_2O_4$ nanoparticles **[A2]**. In addition, the utilization of reduced graphene oxide (rGO) as an additional filler, combined with magnetic spinel ferrites, can contribute to the enhancement of interfacial polarization, enhanced electrical conductivity, and favorable impedance matching. The developed nanocomposites with a thickness of 1 mm demonstrated the highest total shielding effectiveness (SE_T) values of 42.9 dB, 46.2 dB, and 58.8 dB for the CuCoF1-rGO-TPU, CuCoF2-rGO-TPU, and CuF3-rGO-TPU nanocomposites, respectively. The study revealed that 99.9998% of EM waves can be effectively blocked within the tested frequency range. This suggests that the CuF3-rGO-TPU nanocomposite has the potential to serve as a highly efficient material for EMI shielding. The remarkable EMI shielding ability of nanocomposites is primarily attributed to their plentiful interfacial polarization, dipole relaxation, improved impedance matching condition, and the eddy current effect, and natural resonance in the developed polymer nanocomposite. The present research is anticipated to provide valuable insights for the design and development of lightweight and flexible materials that offer outstanding electromagnetic interference (EMI) shielding characteristic **[A2]**.

5.3 Optimization of $CoFe_2O_4$ Nanoparticles and Graphite Fillers to endow Thermoplastic Polyurethane Nanocomposites with Superior Electromagnetic Interference Shielding Performance

This work aimed to construct a range of nanocomposites using a TPU matrix, $CoFe_2O_4$ nanoparticles, and graphite. The fillers' weight percentages and the nanocomposites' thickness were adjusted and optimized to enhance their effectiveness in EMI shielding applications. Graphite has a high degree of versatility, rendering it suitable for deployment in a diverse range of energy-related applications, including batteries, fuel cells, and supercapacitors **[20,21]**. The incorporation of magnetic fillers, when combined with graphite, contributes to the augmentation of electromagnetic (EM) wave absorption by enhancing the magnetic losses, alongside the dielectric and conduction losses within the polymer nanocomposites **[22]**. Cobalt ferrite ($CoFe_2O_4$) has garnered significant attention in the field of electromagnetic wave absorption due to its notable characteristics, including its high magnetocrystalline anisotropy, moderate saturation magnetization at ambient temperature, physical and chemical stability, and high Snoek's limit **[23,24]**. The presence and dispersion of $CoFe_2O_4$ nanoparticles and graphite inside the TPU polymer matrix are anticipated to give rise to the creation of interfaces and defects. These interfaces and defects have the potential to enhance interfacial polarization and further attenuation of EM waves. In addition to the selection of nanofillers and polymer matrix, the EMI shielding effectiveness can be adjusted by manipulating the filler content, loading content (expressed as weight percentage of filler), and the thickness of the nanocomposites **[25]**. The increased amount of filler loading (conductive/magnetic) inside the polymer matrix has the potential to amplify the shielding effectiveness of polymer nanocomposites. However, exceeding a certain threshold in the loading value may adversely impact the mechanical properties of the material. Hence, the attainment of improved electromagnetic interference (EMI) shielding necessitates the appropriate optimization of dielectric and magnetic fillers **[26]**. This study provides a detailed demonstration of the profound impact of the content weight percentage of $CoFe_2O_4$ and graphite in the TPU matrix on electromagnetic interference (EMI) shielding applications. Benefiting from the optimum amount of electrical filler graphite and magnetic filler $CoFe_2O_4$ nanoparticles, the sample with 35 wt% of graphite and 15 wt% $CoFe_2O_4$ nanoparticles in TPU designated as GF15-TPU demonstrated efficient EMI shielding performance of 41.5 dB possessing 99.993 % of the shielding efficiency. The variation of SE_T as a function of frequency with tuned thickness for 1 mm, 2mm, 4 mm and 5 mm, for the developed GF15-TPU nanocomposites is also investigated and was found to be varied from 17.5 dB to 41.5 dB. Enhanced EMI shielding of GF15-TPU nanocomposite is attributed to the enhanced absorption and lesser reflections of EM waves inside the nanocomposite system owing to the synergic dielectric and magnetic losses, eddy current loss, high attenuation, interfacial polarizations, and multiple scattering. This novel and innovative report based on optimized wt% of graphite and $CoFe_2O_4$ nanoparticles in the TPU matrix demonstrates efficient EMI shielding characteristics and can be anticipated for the commercial applications**[A3]**.

6. CONCLUDING REMARKS

6.1 Summary of accomplishments in relation to defined research objectives

The thesis aims at developing advanced spinel ferrite polymer nanocomposite and further exploring its applications for electromagnetic interference shielding. The following is a concise and sequential explanation of the actions required to accomplish the aim mentioned above:

- Identification and selection of the potential magnetic spinel ferrite nanomaterials based on structural and magnetic properties. Further, tailoring the structural, morphological and magnetic characteristics of the nanoparticles by selection of appropriate dopant along with optimized dopant stoichiometric ratio. Spinel ferrite nanoparticles such as cobalt ferrite $CoFe_2O_4$ was selected with various dopant ions including Cu^{2+} and Zn^{2+}. On the basis of the selection of the potential magnetic nanoparticles, sonochemical synthesis route was selected for the successful synthesis of the spinel ferrite nanoparticles considering the purity, good dispersion of the nanomaterials and simplicity of the route.
- Potential conducting filler was selected based on their high electrical conductivity, possessing high dielectric losses and conduction losses. Graphene oxide was synthesized using a modified Hummer's method. Conducting filler rGO was prepared by chemical reduction of synthesized graphene oxide.
- The selection of the suitable potential polymer matrix was chosen on the basis of attributes like flexibility, stretchability, exceptional mechanical capabilities, and resistance to wear. The polymer nanocomposites with spinel ferrite nanoparticles and rGO or graphite were fabricated using melt-mixing approach and further hot-compressed to obtain the desired polymer nanocomposite sheet.
- The developed polymer nanocomposites were comprehensively investigated for structural, morphological, and magnetic characteristics along with characterization of the individual conducting and magnetic fillers. Further, the developed polymer nanocomposites were examined for its EMI shielding properties and electromagnetic parameters including complex permittivity, complex permeability, eddy current losses, attenuation constant and imepdance matching coefficient. All

the EM shielding testing was performed using wave-guide technique in X-band frequency range. The role of dopant ions in the spinel ferrite nanoparticles and presence of secondary phases was evaluated in improving the EMI shielding performance of the developed polymer nanocomposites.

- The fraction loading or amount of conducting and magnetic filler was also optimized for incorporation in the polymer matrix by studying its structural, magnetic and EM properties as well as EMI shielding properties.
- A thickness-based study was also performed to understand the correlation between thickness and absorption of the EM waves in the developed nanocomposites.
- For all the fabricated polymer nanocomposites, an EMI shielding effectiveness much greater than 20 dB was obtained and thereby confirming their suitability for commercial applications. Considering the role of thickness and density of the material, specific shielding effectiveness and absolute shielding effectiveness was also evaluated.
- The dependence of the EM parameters was correlated with the EMI shielding performance of the polymer nancomposites with a possible mechanism.
- The experimental data and results obtained in this dissertation have been published in a high-ranking publication with a Q1/Q2 impact factor, as required by the University Policy and the rector's mandate.

6.2 Summary of research work reports

An overview of the research work that has already been completed and the relevant published work for this doctoral thesis is described below:

***Article I** titled* 'High-Performance, Lightweight, and Flexible Thermoplastic Polyurethane Nanocomposites with Zn^{2+}-Substituted $CoFe_2O_4$ Nanoparticles and Reduced Graphene Oxide as Shielding Materials against Electromagnetic Pollution.'

Main theme of the research work: Designing and fabrication of advanced TPU based nanocomposites by achieving a better balance between the dielectric and magnetic loss to reach the high electromagnetic interference shielding performance along with flexibility, lightness, and thinness. The current study examined the influence of Zn^{2+} substitution on the structural

and magnetic characteristics of $CoFe_2O_4$ spinel ferrite nanoparticles. The substitution of diamagnetic Zn^{2+} ions, , in $CoFe_2O_4$ spinel ferrite can cause changes in the structural (associated to their distribution at tetrahedral and octahedral sites) and electromagnetic properties. The study examined the effect of substituting Zn^{2+} in $CoFe_2O_4$ spinel ferrite nanoparticles on the EMI shielding capabilities of their nanocomposites. TPU-based nanocomposites were fabricated with a composition of 20 wt % nanofillers, consisting of Zn^{2+}-substituted $CoFe_2O_4$ spinel ferrite nanoparticles (CZ1, CZ2, and CZ3) and reduced graphene oxide (rGO) in a 9:1 wt % ratio. The nanoparticles and their nanocomposites were examined in depth to determine their structural, morphological, and electromagnetic properties. The nanocomposites with a thickness of 0.8 mm achieved maximum total shielding effectiveness (SE_T) values of 48.3 dB, 61.8 dB, and 67.8 dB for CZ1-rGO-TPU, CZ2-rGO-TPU, and CZ3-rGO-TPU, respectively, in the frequency range of 8.2–12.4 GHz. The $Co_{0.33}Zn_{0.67}Fe_2O_4$ (CZ3)-rGO-TPU nanocomposite exhibits excellent electromagnetic interference shielding properties due to dipole and interfacial polarization, conduction loss, multiple scattering, eddy current effect, natural resonance, high attenuation constant, and impedance matching. The obtained results concluded that the optimized CZ3-rGO-TPU nanocomposite has the potential to serve as a lightweight, flexible, thin, and high-performance material for shielding against EMI.
Contribution: writing original draft, investigation, experimental, and editing.

Article II *titled* ‘$Cu_xCo_{1-x}Fe_2O_4$ (x = 0.33, 0.67, 1) Spinel Ferrite Nanoparticles Based Thermoplastic Polyurethane Nanocomposites with Reduced Graphene Oxide for Highly Efficient Electromagnetic Interference Shielding’

Main theme of the research work: This study employed hybrid filler systems consisting of $Cu_xCo_{1-x}Fe_2O_4$ (x = 0.33, 0.67, 1) spinel ferrite nanoparticles and rGO embedded within a TPU matrix. The magnetic hysteresis curves of CuCoF1, and CuCoF2 show typical ferromagnetic features whereas the magnetic hysteresis curve of CuF3 display an S-shape with no coercivity and remanence, which points to the superparamagnetic characteristic, and can be beneficial for utilization as high-performance electromagnetic interference shielding material at high frequency. The developed nanocomposites with a thickness of 1 mm, showed the

highest total shielding effectiveness (SE_T) values of 42.9 dB, 46.2 dB, and 58.8 dB for the CuCoF1-rGO-TPU, CuCoF2-rGO-TPU, and CuF3-rGO-TPU nanocomposites, respectively. The study revealed that 99.9998% of EM waves can be effectively blocked within the tested frequency range. This suggests that the CuF3-rGO-TPU nanocomposite is a very efficient material for electromagnetic shielding. The excellent EMI shielding ability of nanocomposites is primarily attributed to their plentiful interfacial polarization, dipole relaxation, improved impedance matching condition, and the eddy current effect. This research work yield valuable insights for the development of lightweight and flexible shielding materials that have exceptional electromagnetic interference (EMI) shielding capabilities.
Contribution: formal analysis, visualization, investigation, writing-original draft.

Article III *titled* 'Optimization of $CoFe_2O_4$ Nanoparticles and Graphite Fillers to Endow Thermoplastic Polyurethane Nanocomposites with Superior Electromagnetic Interference Shielding Performance.'

Main theme of the research work: Within this study, deep insight into the influence of content wt.% of $CoFe_2O_4$ nanoparticles and further optimization of the wt% of the fillers in the TPU matrix for EMI shielding applications is demonstrated comprehensively. Combining magnetic materials with electrical materials is an effective strategy to improve EM wave absorption by achieving balanced impedance matching and attenuation of EM waves. A proper optimization of dielectric and magnetic fillers is a prerequisite for achieving enhanced EMI shielding. To develop the nanocomposite along with dielectric and magnetic properties, varying content wt.% of $CoFe_2O_4$ nanoparticles and graphite were embedded in the TPU matrix. A comprehensive analysis was conducted to assess the structural and morphological properties of both the $CoFe_2O_4$ nanoparticles and the nanocomposites. To conduct a thorough examination, a detailed investigation of the magnetic properties curve was performed for both the $CoFe_2O_4$ nanoparticles and the nanocomposites. Benefiting from the optimum amount of electrical filler graphite and magnetic filler $CoFe_2O_4$ nanoparticles, the sample with 35 wt.% of graphite and 15 wt.% $CoFe_2O_4$ nanoparticles in TPU designated as GF15-TPU demonstrated an enhanced EMI shielding performance of 41.5 dB possessing 99.993 % of the shielding efficiency with an improvement in the absorption of EM waves. Enhancing EMI shielding is attributed to synergic dielectric and magnetic losses, eddy current loss, high attenuation, interfacial polarizations, and multiple scattering.

Contribution: methodology; data curation; formal analysis; writing - original draft; investigation.

6.3 Contribution to Science, practical implication, and future directions

The main objective of this PhD research was focused on the development of advanced spinel ferrite polymer nanocomposites for EM application. The goals were successfully accomplished by the preparation of novel innovative spinel ferrite based polymer nanocomposites along with rGO or graphite as a secondary filler in TPU matrix. All the developed polymer nanocomposites demonstrated superior EM shielding performance and were able to effectively attenuate the EM waves alongwith the major contribution from the absorption of EM waves and therefore reducing secondary reflections. All of the developed polymer nanocomposites displayed value of SE_T >20 dB and therefore making them as a suitable candidate for commercial applications. Additionally, the developed polymer nanocomposites were flexible, thin, and lightweight making it convenient for applications in X-band frequency range which includes application in aircraft, satellite, telecommunications, and in the field of radar stealth.

Nevertheless, the present research primarily focuses on limited-scale investigations lacking extended real-world implementations. Based on the present research trends and anticipated future research direction, it is possible to establish the following primary goals for future work:

- Development of heterostructured nanocomposites such as graphene@spinel ferrites with more interfaces and their utilization in suitable polymer matrix and further study of its EMI shielding properties.
- Exploring other secondary conducting fillers such as MXene $Ti_3C_2T_x$ and graphene quantum dots for incorporation in the polymer matrix for EMI shielding applications.
- The aim will be to cover the whole broad-band frequency range (2-18 GHz) by the developed nanocomposite for expanding the application

area of the developed nanocomposite to be suitable for applications in aircraft, submarines, automobiles, flexible electronics, etc.

- The focus will be on the development of EMI shielding materials based on utilization of waste from industrial magnet production to make the process cost-effective.

References from Author's works

[A1]: **Anju**, R.S. Yadav, P. Pötschke, J. Pionteck, B. Krause, I. Kuřitka, J. Vilcakova, D. Skoda, P. Urbánek, M. Machovsky, M. Masař, M. Urbánek, M. Jurca, L. Kalina, J. Havlica, High-Performance, Lightweight, and Flexible Thermoplastic Polyurethane Nanocomposites with Zn^{2+}-Substituted $CoFe_2O_4$ nanoparticles and Reduced Graphene Oxide as Shielding Materials against Electromagnetic Pollution, ACS Omega 6 (2021) 28098-28118. https://doi.org/10.1021/ACSOMEGA.1C04192.

[A2]: **Anju**, R.S. Yadav, P. Pötschke, J. Pionteck, B. Krause, I. Kuřitka, J. Vilčáková, D. Škoda, P. Urbánek, M. Machovský, M. Masař, M. Urbánek, $Cu_xCo_{1-x}Fe_2O_4$ (x = 0.33, 0.67, 1) Spinel Ferrite Nanoparticles Based Thermoplastic Polyurethane Nanocomposites with Reduced Graphene Oxide for Highly Efficient EMI Shielding, Int. J. Mol. Sci. 23 (2022) 2610. https://doi.org/10.3390/IJMS23052610.

[A3]: **Anju**, M. Masař, M. Machovský, M. Urbánek, P. Šuly, B. Hanulíková, J. Vilčáková, I. Kuřitka, R.S. Yadav, Optimization of $CoFe_2O_4$ Nanoparticles and Graphite Fillers to endow Thermoplastic Polyurethane Nanocomposites with Superior Electromagnetic Interference Shielding Performance. Nanoscale Advances, 6 (2024) 2149-2165; Doi: https://doi.org/10.1039/D3NA01053H.

[A4]: Raghvendra Singh Yadav, **Anju** and Ivo Kuritka, Spinel ferrite and MXene-based magnetic novel nanocomposites: an innovative high-performance electromagnetic interference shielding and microwave absorber, Critical Reviews in Solid State and Materials Sciences 48(4) (2023) 441–479; Doi: 10.1080/10408436.2022.2067122.

REFERENCES

[1] C.M. Koo, P. Sambyal, A. Iqbal, F. Shahzad, J. Hong, Two-Dimensional Materials for Electromagnetic Shielding, 2021. https://doi.org/10.1002/9783527829828.

[2] J. Li, E. Masghouni, M. Granger, G. Sudre, P. Alcouffe, D. Muller, V.S. Nguyen, B. Bayard, A. Serghei, B. Sauviac, A. Maazouz, K. Lamnawar, Design, Processing, and Challenges of Multicomponent Polymer Composites for Improved Electromagnetic Interference Shielding Properties: A Review, Macromol Mater Eng (2024). https://doi.org/10.1002/MAME.202300344.

[3] S. Thomas, S.M. Zachariah, Nanostructured Materials for Electromagnetic Interference Shielding, CRC Press, Boca Raton, 2021. https://doi.org/10.1201/9781003217312.

[4] M. Aghvami-Panah, A. Ameli, MXene/Cellulose composites as electromagnetic interference shields: Relationships between microstructural design and shielding performance, Compos Part A Appl Sci Manuf 176 (2024) 107879. https://doi.org/10.1016/J.COMPOSITESA.2023.107879.

[5] L. Li, Y. Cao, X. Liu, J. Wang, Y. Yang, W. Wang, Multifunctional MXene-Based Fireproof Electromagnetic Shielding Films with Exceptional Anisotropic Heat Dissipation Capability and Joule Heating Performance, ACS Appl Mater Interfaces 12 (2020) 27350–27360. https://doi.org/10.1021/acsami.0c05692.

[6] D. Xiang, X. Zhang, Y. Li, E. Harkin-Jones, Y. Zheng, L. Wang, C. Zhao, P. Wang, Enhanced performance of 3D printed highly elastic strain sensors of carbon nanotube/thermoplastic polyurethane nanocomposites via non-covalent interactions, Compos B Eng 176 (2019) 107250. https://doi.org/10.1016/J.COMPOSITESB.2019.107250.

[7] M. Panahi-Sarmad, M. Noroozi, X. Xiao, C.B. Park, Recent Advances in Graphene-Based Polymer Nanocomposites and Foams for Electromagnetic Interference Shielding Applications, Ind Eng Chem Res 61 (2022) 1545–1568. https://doi.org/10.1021/ACS.IECR.1C04116.

[8] S. Hema, S. Sambhudevan, Ferrite-based polymer nanocomposites as shielding materials: a review, Chemical Papers 75 (2021) 3697–3710. https://doi.org/10.1007/s11696-021-01664-1.

[9] R.S. Yadav, I. Kuřitka, Recent developments on nanocomposites based on spinel ferrite and carbon nanotubes for applications in electromagnetic interference shielding and microwave absorption, Critical Reviews in Solid State and Materials Sciences (2023). https://doi.org/10.1080/10408436.2023.2214577.

[10] S.B. Narang, K. Pubby, Nickel Spinel Ferrites: A review, J Magn Magn Mater 519 (2021) 167163. https://doi.org/10.1016/J.JMMM.2020.167163.

[11] M. Panahi-Sarmad, M. Noroozi, X. Xiao, C.B. Park, Recent Advances in Graphene-Based Polymer Nanocomposites and Foams for Electromagnetic Interference Shielding Applications, Ind Eng Chem Res 61 (2022) 1545–1568. https://doi.org/10.1021/acs.iecr.1c04116.

[12] A.D. Jara, A. Betemariam, G. Woldetinsae, J.Y. Kim, Purification, application and current market trend of natural graphite: A review, Int J Min Sci Technol 29 (2019) 671–689. https://doi.org/10.1016/j.ijmst.2019.04.003.

[13] D.D.L. Chung, Review: Graphite, J Mater Sci 37 (2002) 1475–1489. https://doi.org/10.1023/A:1014915307738.

[14] Y. Chen, S. Li, S. Lin, M. Chen, C. Tang, X. Liu, Promising energy-storage applications by flotation of graphite ores: A review, Chemical Engineering Journal 454 (2023) 139994. https://doi.org/10.1016/J.CEJ.2022.139994.

[15] E. George, J. Joy, S. Anas, Acrylonitrile-based polymer/graphene nanocomposites: A review, Polym Compos 42 (2021) 4961–4980. https://doi.org/10.1002/PC.26224.

[16] A. Jiříčková, O. Jankovský, Z. Sofer, D. Sedmidubský, Synthesis and Applications of Graphene Oxide, Materials 15 (2022). https://doi.org/10.3390/ma15030920.

[17] M. Ramezani, M.Alibolandi, M. Nejabat, F. Charbgoo, S. M.Taghdisi, K.Abnous, Graphene-Based Hybrid Nanomaterials for Biomedical Applications. Biomedical Applications of Graphene and 2D Nanomaterials, Micro and Nano Technologies, (2019) 119–141. https://doi.org/10.1016/B978-0-12-815889-0.00006-4.

[18] M. Thangamuthu, K.Y. Hsieh, P. V. Kumar, G.Y. Chen, Graphene- and graphene oxide-based nanocomposite platforms for electrochemical biosensing applications, Int J Mol Sci 20 (2019). https://doi.org/10.3390/ijms20122975.

[19] T.M. Hammad, S. Kuhn, A.A. Amsha, R. Hempelmann, Investigation of structural, optical, and magnetic properties of Co^{2+} ions substituted $CuFe_2O_4$ spinel ferrite nanoparticles prepared via precipitation approach, Journal of the Australian Ceramic Society 57 (2021) 543–553. https://doi.org/10.1007/S41779-020-00556-Z/FIGURES/13.

[20] S.E. Berrabah, A. Benchettara, F. Smaili, A. Benchettara, A. Mahieddine, High performance hybrid supercapacitor based on electrochemical deposed of nickel hydroxide on zinc oxide supported by graphite electrode, J Alloys Compd 942 (2023) 169112. https://doi.org/10.1016/j.jallcom.2023.169112.

[21] D. Zhang, W. Zhang, S. Zhang, X. Ji, L. Li, Synthesis of expanded graphite-based materials for application in lithium-based batteries, 60 (2023). https://doi.org/10.1016/j.est.2023.106678.

[22] R.K. Bheema, K.C. Etika, Large microwave absorption By Fe_3O_4@CuNW hybrid nanoparticles filled epoxy nanocomposites in the X-Band, J Alloys Compd 938 (2023) 168405. https://doi.org/10.1016/j.jallcom.2022.168405.

[23] F.E. Carvalho, L. V. Lemos, A.C.C. Migliano, J.P.B. Machado, R.C. Pullar, Structural and complex electromagnetic properties of cobalt ferrite ($CoFe_2O_4$) with an addition of niobium pentoxide, Ceram Int 44 (2018) 915-921. https://doi.org/10.1016/J.CERAMINT.2017.10.023.

[24] Y. Liu, Z. Chen, Y. Zhang, R. Feng, X. Chen, C. Xiong, L. Dong, Broadband and Lightweight Microwave Absorber Constructed by in Situ Growth of Hierarchical $CoFe_2O_4$/Reduced Graphene Oxide Porous Nanocomposites, ACS Appl Mater Interfaces 10 (2018) 13860–13868. https://doi.org/10.1021/ACSAMI.8B02137.

[25] D. Jiang, V. Murugadoss, Y. Wang, J. Lin, T. Ding, Z. Wang, Q. Shao, C. Wang, H. Liu, N. Lu, R. Wei, A. Subramania, Z. Guo, Electromagnetic Interference Shielding Polymers and Nanocomposites

- A Review, Polymer Reviews 59 (2019) 280–337. https://doi.org/10.1080/15583724.2018.1546737.

[26] X. Li, L. Yu, W. Zhao, Y. Shi, L. Yu, Y. Dong, Y. Zhu, Y. Fu, X. Liu, F. Fu, Prism-shaped hollow carbon decorated with polyaniline for microwave absorption, Chemical Engineering Journal 379 (2020) 122393. https://doi.org/10.1016/j.cej.2019.122393.

LIST OF FIGURES

LIST OF ABBREVIATIONS AND SYMBOLS

EMI	Electromagnetic interference
SE_M	Shielding due to multiple reflections
SE_R	Shielding due to reflections
SE_A	Shielding due to absorption
SE_T	Total shielding effectiveness
$tan\delta_\varepsilon$	Dielectric loss

$tan\delta_\mu$	Magnetic loss
C_o	Eddy current loss
γ	gyromagnetic ratio
H_a	Anistropy energy
α	Attenuation constant
Z	Impedance matching coefficient
SSE	Specific shielding effectiveness
GO	Graphene oxide
rGO	reduced-Graphene oxide
TPU	Thermplastic Polyurethane
μ''	imaginary part of permeability
μ'	real part of permeability
σ_{ac}	electrical conductivity
A_{eff}	Effective Absorption
δ	Skin depth
SSE_t	Absolute shielding effectiveness
M_s	Saturation magnetization
M_r	Remanant magnetization
H_c	Coercivity
$\lvert K_1 \rvert$	Anisotropic coefficient
f_{exc}	Exchange frequency

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- Member of the research team (2020): IGA/CPS/2020/003- "Preparation and characterization of nanoparticles for advanced applications".
- Member of the research team (2021): IGA/CPS/2021/002 - "Preparation and characterization of nanocomposite systems".
- Member of the research team (2022): IGA/CPS/2022/002 - "Preparation and characterization of advanced nanocomposite systems".

- Member of the research team (2023): IGA/CPS/2023/006 - "Preparation and characterization of advanced functional nanocomposite systems".
- Member of the research team (2024): IGA/CPS/2024/002 - "Study of the preparation of nanoparticles using oxidized polysaccharides".

External funding projects:

- Research and development of solar reflexive coating system for enhancing the energy performance of buildings (FW06010191) - Research Project staff.
- Ministry of Education, Youth, and Sports of the Czech Republic - DKRVO (RP/CPS/2022/007)- Research Project staff.
- Project Grantová agentura České republiky (GAČR) 19-23647S (2020-2021) -Research on the Correlation between Cation Distribution, Particle Size and Physical Properties of Smart Spinel-Ferrite Nanomaterials, Research Project Staff.

List of Publications

- **Anju**, Milan Masař, Michal Machovský, Michal Urbánek, Pavol Šuly, Barbora Hanulíková, Jarmila Vilčáková, Ivo Kuřitka, Raghvendra Singh Yadav, *Optimization of $CoFe_2O_4$ Nanoparticles and Graphite Fillers to Endow Thermoplastic Polyurethane Nanocomposites with Superior Electromagnetic Interference Shielding Performance*, Nanoscale Adv., 2024,6, 2149-2165; Doi: https://doi.org/10.1039/D3NA01053H.
- **Anju**, Raghvendra Singh Yadav, Petra Potschke, Jürgen Pionteck, Beate Krause, Ivo Kuritka, Jarmila Vilcakova, David Skoda, Pavel Urbanek, Michal Machovsky, Milan Masar and Michal Urbanek, *$Cu_xCo_{1-x}Fe_2O_4$ (x = 0.33, 0.67, 1) Spinel Ferrite Nanoparticles Based Thermoplastic Polyurethane Nanocomposites with Reduced Graphene Oxide for Highly Efficient Electromagnetic Interference Shielding*, Int. J. Mol. Sci. 2022, 23(5), 2610; Doi: https://doi.org/10.3390/ijms23052610.
- **Anju**, Raghvendra Singh Yadav, Petra Pötschke, Jurgen Pionteck, Beate Krause, Ivo Kuritka, Jarmila Vilcakova, David Skoda, Pavel Urbanek, Michal Machovsky, Milan Masar, Michal Urbanek, Marek Jurca, Lukas Kalina, and Jaromir Havlica, *High-Performance, Lightweight, and Flexible Thermoplastic Polyurethane*

Nanocomposites with Zn^{2+}-Substituted $CoFe_2O_4$ Nanoparticles and Reduced Graphene Oxide as Shielding Materials against Electromagnetic Pollution, ACS Omega 2021, 6(42), 28098–28118. Doi: https://doi.org/10.1021/acsomega.1c04192.

- Raghvendra Singh Yadav, **Anju** and Ivo Kuritka, *Spinel ferrite and MXene-based magnetic novel nanocomposites: an innovative high-performance electromagnetic interference shielding and microwave absorber*, Critical Reviews in Solid State and Materials Sciences 2022, 48(4), 441–479. Doi: 10.1080/10408436.2022.2067122.
- Raghvendra Singh Yadav, **Anju**, Thaiskang Jamatia, Ivo Kuritka, Jarmila Vilcakova, David Skoda, Pavel Urbanek, Michal Machovsky, Milan Masar, Michal Urbanek, Lukas Kalina, and Jaromir Havlica, *Superparamagnetic $ZnFe_2O_4$ Nanoparticles-Reduced Graphene Oxide-Polyurethane Resin Based Nanocomposites for Electromagnetic Interference Shielding Application*, Nanomaterials 2021, *11*(5), 1112; Doi: https://doi.org/10.3390/nano11051112.
- Raghvendra Singh Yadav, **Anju**, Thaiskang Jamatia, Ivo Kuritka, Jarmila Vilcakova, David Skoda, Pavel Urbanek, Michal Machovsky, Milan Masar, Michal Urbanek, Lukas Kalina and Jaromir Havlica, *Excellent, Lightweight and Flexible Electromagnetic Interference Shielding Nanocomposites Based on Polypropylene with $MnFe_2O_4$ Spinel Ferrite Nanoparticles and Reduced Graphene Oxide*, Nanomaterials 2020, 10(12), 2481; Doi: https://doi.org/10.3390/nano10122481.
- **Anju**, Anam Rais, Kamla Rawat, Tulika Prasad, and H B Bohidar, *Boron-doped carbon quantum dots: a 'turn-off' fluorescent probe for dopamine detection*, Nanotechnology 2020, 32025501 (9pp), https://doi.org/10.1088/1361-6528/abb84d.

Conference Participation and proceedings

- **Anju**, Raghvendra Singh Yadav, Ivo Kuritka, Jarmila Vilcakova, David Skoda, Pavel Urbanek, Michal Machovsky, Milan Masar, Michal Urbanek; *Lightweight, Flexible and High-Performance nanocomposites based on Reduced Graphene Oxide and spinel ferrite ($ZnFe_2O_4$ / $CoFe_2O_4$) nanoparticles in Thermoplastic Polyurethane Matrix for Electromagnetic Interference Shielding Applications*: Proceedings 13th International Conference on Nanomaterials -

Research & Application, Brno, Czech Republic, DOI : https://doi.org/10.37904/nanocon.2021.4323**.**

- Poster Presentation on '*Electromagnetic Interference Shielding Performance of Thermoplastic Polyurethane based nanocomposites filled with $CoFe_2O_4$ nanoparticles alongwith Graphite for X-band Frequency range*: PolyScience 2023: 3rd Global Summit on Polymer Science and Composite Materials (PolyScience2023) in Lisbon, Portugal.

Other Scientific Activities

Edited Book:

- Raghvendra Singh Yadav, **Anju** and Kottakkaran Sooppy Nisar, Structural, Magnetic, Dielectric, Electrical, Optical and Thermal Properties of Nanocrystalline Materials: Synthesis, Characterization and Application, crystals, MDPI, ISBN 978-3-0365-3155-7.

Guest co-editor for Crystal (MDPI, Switzerland)

- Special Issue "Structural, Magnetic, Dielectric, Electrical, Optical and Thermal Properties of Nanocrystalline Materials: Synthesis, Characterization and Application", (Volume I) Crystals, MDPI.
- Special Issue "Structural, Magnetic, Dielectric, Electrical, Optical and Thermal Properties of Nanocrystalline Materials: Synthesis, Characterization and Application", (Volume II) Crystals, MDPI.

Anju, M.Sc., Ph.D.

Preparation and characterization of advanced spinel ferrite nanocomposites for electromagnetic applications

Příprava a charakterizace pokročilých spinel feritových nanokompozitů pro elektromagnetické aplikace

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