# Microwave absorption properties of Fe-Co oxide - thermally exfoliated graphene oxide composite

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# Abstract

In the ongoing search for newer materials for microwave absorption applications, the graphene merits a special consideration owing to its outstanding mechanical, thermal and electrical properties. Here, we report investigation on microwave absorption properties of Fe-Co oxide- thermally exfoliated graphene oxide (Fe-Co oxide-TEGO) composite in a frequency range of 8-18 GHz. Fe-Co oxide was synthesized by thermal decomposition of Fe-Co oxide/aniline- formaldehyde copolymer at 900 °C in air. Fe-Co oxide-TEGO hybrid was prepared by physical mixing of Fe-Co oxide and TEGO. Thereafter, hybrid was characterized using scanning electron microscope, energy dispersive X-ray, Fourier transform infrared spectrometer and X-ray diffractrometer. Thereafter, Fe-Co oxide-TEGO composite on electromagnetic properties were studied. The effect of varying the weight % of Fe-Co oxide in Fe-Co oxide-TEGO composite on electromagnetic properties was also studied. The absorption bandwidth of 3.5 GHz (8.5-12.0 GHz, X-band) and 5.0 GHz (13.0-18.0 GHz, Ku -band) has been achieved using Fe-Co oxide-TEGO composite (50 wt%) with 2.2 and 1.6 mm thickness, respectively. Microwave absorption studies show Fe-Co oxide-TEGO composite as a potential candidate for microwave absorption applications. Copyright© 2018 VBRI Press.

Keywords: Graphite oxide, composite, dielectric loss, microwave absorption, lightweight absorber.

#### Introduction

In recent years, the development of microwave absorbing materials has attracted much attention for electromagnetic interference (EMI) shielding and electromagnetic (EM) wave absorbing applications, for both commercial and defence purposes [1–3]. The key requirement in this field is the preparation of microwave absorbers that have wide absorption frequency range, small thickness, light weight and strong absorption characteristics [4-8]. Traditional magnetic EM wave absorption materials such as spineltype ferrites [9,10] and magnetic metal powders [11,12] exhibit large magnetic loss due to their high complex permeability. However, the magnetic absorbers have relatively higher densities thus suffer from more weight penalty, which restrains their practical application [13]. On the other hand, dielectric absorbers have the advantages such as low density, controllable dielectric loss ability and so on [14]. Recently, many studies have been carried out to investigate the complementary behaviour of magnetic or dielectric microwave absorber coatings by mixing multi-walled carbon nanotubes or metallic magnetic materials [15-20]. CNTs however, are economically non-viable, difficult to produce in bulk and require purification, auxiliary treatment, and functionalization steps [**21**, **22**]. Therefore, designing and fabricating new type of EM wave absorption materials are highly desirable.

Graphene, a novel two-dimensional carbon material, on the other hand has triggered enormous interest due to its fascinating electrical properties [23, 24]. Graphene possesses not only a stable structure but also high-specific surface area and excellent electronic conductivity. These properties make graphene or graphene-based materials very promising as a new type of strong and lightweight EM wave absorption materials [25]. However, the study finds out that the good electric conductivity of pure graphene becomes a disadvantageous factor according to the impedance match mechanism [26]. Decorating graphene with inorganic nanoparticles is an available method to form graphene-based hybrid, and the combination of inorganic nanoparticles and graphene can result in some tailored EM properties. Magnetic graphene based hybrids as lightweight microwave absorption materials have drawn extensive attention such as Co, Fe, Fe<sub>3</sub>O<sub>4</sub>, carbonyl iron, etc. It is reported that the α-Co nanocrystals/GN nano-composites exhibit excellent electromagnetic performance [27]. Zhao et al investigated microwave absorption properties of graphene-coated Fe nanocomposites [28]. He et al. reported a facile solvothermal route to prepare laminated TEGO/Fe<sub>3</sub>O<sub>4</sub> composites, with reflection loss below-10 dB at 2 GHz and a maximum absorption of -26.4 dB [29]. Zhang et al. reported a maximum absorption of -33.5 dB from a TEGO/  $\alpha$  -Fe<sub>2</sub>O<sub>3</sub> composite hydrogel prepared via a twostep process [31]. Yin et al. fabricated TEGO/  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> composite with reflection loss of -59.65 dB at 10.09 GHz [32]. RGO-CoFe<sub>2</sub>O<sub>4</sub> composite synthesized by Zong et al found to show the absorption bandwidth (reflection loss >10 dB) of 4.7 GHz (from 13.3 to 18.0 GHz) with a thickness of 1.5 mm [33].

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This study aims to develop a facile approach to fabricate high-performance magnetic materials/TEGO hybrid microwave absorbers, as well as to investigate any synergistic interaction between the magnetic materials and TEGO. Herein, we have prepared Fe-Co oxide/TEGO hybrid by physical mixing of TEGO and Fe-Co oxide and investigated their EM and microwave absorption properties for practical applications in the future.

# Experimental

## Materials

Aniline, formaldehyde solution (37-41% w/v), cobaltous chloride hexahydrate, sodium hydroxide, hydrochloric acid, ferric chloride (anhydrous) were analytical grade and procured from Merck, India and used to synthesise Fe-Co oxide nanocomposites in aniline-formaldehyde copolymer matrix. Graphite powder (mesh size 50 micron), sulfuric acid (assay, 98.0%), phosphoric acid (assay, 85%), sodium nitrate (99% GR), potassium permanganate (99% GR), hydrogen peroxide (30%). All chemicals were procured from Merck, India and used directly without further purification to synthesize graphite oxide. Polyurethane resin (M/s Synpol Products Pvt. Ltd., India) was used as matrix for composite preparation.

# Synthesis of Fe-Co oxide-aniline formaldehyde nanocomposite

Fe-Co oxide-aniline formaldehyde nanocomposite was synthesized by co-precipitation of oxides of Fe and Co by dissolving ferric chloride and cobaltous chloride hexahydrate in 7:3 molar ratio followed by its addition into aniline-formaldehyde copolymer matrix followed by adding alkali solution (10 %) to the mixture with vigorous stirring. Thereafter, the mixture was vacuum filtered and dried in oven at 110  $^{\circ}$ C for 4h [**34**].

# Preparation of graphite oxide and TEGO

Graphite oxide (GO) was synthesized from graphite powder using modified Hummer's method [**35**]. In a typical preparation, 6 g of sodium nitrate were mixed into 230 ml of conc. sulphuric acid and phosphoric acid (9:1 weight ratio) under constant stirring followed by the addition of 10 g of graphite. After 4 h, 30 g of KMnO<sub>4</sub> was added gradually to the above solution while keeping the temperature less than 20°C to prevent overheating and explosion. The mixture was stirred at 35 °C for 48 h and the resulting solution was diluted by adding 460 ml of water under vigorous stirring. To ensure the completion of reaction with KMnO<sub>4</sub>, the suspension was further treated with 30%  $H_2O_2$  solution (20 ml). The resulting mixture was washed with HCl and  $H_2O$  respectively, followed by filtration and drying, GO were thus obtained. The graphite oxide was thermally treated at 300 °C in air at atmospheric pressure for 10 min. The GO got exfoliated after thermal treatment and results into partial reduction of graphene oxide.

# Preparation of Fe- Co oxide-TEGO /PU composite

The calculated amount of Fe-Co oxide and TEGO were physically mixed and dispersed into PU resin and the mixture was blended by a shear mixer to make a homogeneous blend, thereafter, a curing agent was further added into the blend. Finally, the mixture was poured into the mold and cured at room temperature. After curing, composite sheet was carefully removed from the mould. Three composites were prepared for EM characterization. The composition of composites is given in **Table 1.** 

 Table 1. Composition details of composites.

Sample	TEGO [wt%]	Fe-Co Oxide [wt%]	PU [wt%]
TEGO: 10 %	10	Nil	90
Fe-Co Oxide – TEGO: 40%	10	30	60
Fe-Co Oxide – TEGO: 50%	10	40	50

### **Characterizations**

Scanning Electron Microscope (SEM, Carl Zeiss SMT Ltd, EVO-MA 15) equipped with an energy dispersion Xray spectroscope (EDX, INCA Energy 250 Microanalysis System) was used to analyze the surface morphology at operating voltage of 20 kV, while EDX analysis was performed to understand their chemical constituents. The Fourier transform infrared (FTIR) spectra were recorded in KBr with a resolution of 2 cm<sup>-1</sup> and scan range of 400-4000 cm<sup>-1</sup> by using ATR-FTIR spectrophotometer, Agilent; Model: 660. Pellets were formed after pressing in hydraulic press at about 1.5 tonne/cm<sup>2</sup> for the FTIR measurements. The structural characterization of the samples was carried out by the powder X-ray diffractometer (Philips X'Pert Pro diffractometer) using  $CuK\alpha$  ( $\lambda = 0.154$  nm) radiation source operated at the voltage of 40kV and current of 30mA over the scattering angle ranging from  $2\Theta=5-80^{\circ}$  with a step size of 0.05<sup>0</sup>/min. Vector network analyzer (VNA, model Hewlett-Packard 8510) was used to obtain S-parameters of the samples in the 8-12 GHz range at room temperature. EM parameters (complex permittivity and

complex permeability) of the TEGO and Fe- Co oxide-TEGO/PU composites were calculated from the measured values of S-parameters using Nicolson-Ross algorithm [**36**]. The microwave absorbing properties of a one-layer absorber backed by a metallic plate were then investigated by means of numerical simulations considering a plane wave incidence on the dielectric stratification and a transmission line approach [**37**].

### **Results and discussion**

### Physiochemical characterization

SEM images of TEGO and Fe- Co oxide-TEGO hybrid are shown in **Fig. 1(a) and (b)**. SEM images indicate the layered morphology of TEGO (**Fig. 1(a**)) while the SEM image of Fe- Co oxide-TEGO hybrid, shows the presence of cubic shaped particles of Fe- Co oxide in addition to the graphenic layers (**Fig. 1(b). Fig. 1(c**)) shows the cross sectional view of Fe- Co oxide-TEGO/ PU composite. A uniform dispersion can be seen in the composite. Fe- Co oxide/TEGO hybrid has been subjected to EDX analysis (**Fig. 2**) in order to study and identify the elemental composition which indicates the presence of C, O, Fe and Co.





Fig. 1. SEM images of (a) TEGO (b) Fe-Co oxide-TEGO and (c) Fe-Co oxide-TEGO /PU composite.



Fig. 2. EDX spectra of Fe-Co oxide-TEGO.



Fig. 3. FTIR spectra of TEGO and Fe-Co oxide-TEGO.

FTIR was recorded as shown in **Fig. 3**, to investigate the presence of oxygen-containing functional groups on TEGO and oxide of Fe- Co. After thermal reduction of GO, the peak intensities of C=O (1732 cm<sup>-1</sup>), C-OH (1402 cm<sup>-1</sup>), and epoxy (1224 cm<sup>-1</sup>) groups decreased, the absorbance bands at C-O of alkoxy groups (1047 cm<sup>-1</sup>), had completely vanished and peak at 1577 cm<sup>-1</sup> found to be more pronounced indicating the thermal reduction of GO to TEGO. Peak intensity of adsorbed water molecules at 3408 cm<sup>-1</sup> get reduced after thermal exfoliation of GO. The FTIR spectra Fe- Co oxide/TEGO hybrid was observed similar to TEGO with additional peak at ~590 cm<sup>-1</sup> which is attributed to the M-O (metal-oxide) bond vibration of Fe-Co oxide [**38**].

XRD spectra of TEGO and Fe-Co oxide-TEGO are shown in Fig 4. The diffraction peaks at 25°, 42.2°, 49.1°, and 72.8° in TEGO are attributed to the hexagonal graphite structure planes {002}, {100}, {004}, and {110}. These diffraction peaks are signature of the hexagonal graphite lattice of graphene material and is consistence with the reported results for reduced graphene oxide by our group [39, 40]. The {002} and  $\{004\}$  peaks at 25° and 49.1° correspond to parallel graphene layers. The {100} and {110} diffraction peaks at 42. 2° and 72.8° characterize the 2D in-plane symmetry along the graphene layers. Fe-Co oxide-TEGO shows diffraction peaks at 30.4°, 35.7°,44.0°, 57.3° and 62.8° which are attributed to the  $\{220\}, \{311\}, \{400\}, \{511\}$ and {440} reflections of inverse spinel structure of Fe-Co metal oxide in addition to the diffraction peaks of TEGO [38].

#### Microwave absorption properties

The EM parameters (relative complex permittivity,  $\varepsilon_r = \varepsilon' - j\varepsilon''$  and relative complex permeability,  $\mu_r = \mu' - j\mu''$ ) of the PU composites containing 10 wt % of the pure TEGO and Fe-Co oxide- TEGO hybrids with 40 and 50 wt % in the resultant composites. Fig. 5 (a &b) shows the measured  $\varepsilon'$  (real part) and  $\varepsilon''$  (imaginary part) values of  $\varepsilon_r$  in the range of 8–12 GHz for the composites. As shown in Fig. 5 (a) the  $\varepsilon'$  values of pure TEGO-10 % composite is in the range of 7.9-7.4 and for Fe-Co oxide-TEGO: 40% and Fe-Co oxide-TEGO: 50% composites, the values decrease from 8.8 and 11.5 to 8.4 and 10.5 respectively with increasing frequency in the 8–12 GHz range. The decrease of  $\varepsilon'$  with increase in frequency may arise due to lags of induced charges in the material to follow the reversing electromagnetic filed in the high frequency range. It should be noted that with increase of Fe-Co oxide concentration in the Fe-Co oxide/TEGO PU composites, the  $\varepsilon'$  values increases. The  $\varepsilon''$  values of the samples are shown in **Fig. 5(b)**. The  $\varepsilon''$  values of TEGO-10% composite vary from 2.8 to 2.3 while the values of Fe-Co oxide-TEGO: 40 wt% vary in the range of 2.4 to 2.3 and for Fe-Co oxide-TEGO: 50 wt%, the value varies between 3.8-3.3. It can be seen that the values of  $\varepsilon''$  decreases slightly with the increase in frequency.



Fig. 4. XRD spectra of TEGO and Fe-Co oxide-TEGO.



**Fig. 5.** Frequency dependence on (a) real part of complex permittivity, (b) imaginary part of complex permittivity of TEGO and Fe-Co oxide-TEGO- 40% and Fe-Co oxide-TEGO- 50% composites.



**Fig. 6.** Frequency dependence on (a) real part of complex permeability, (b) imaginary part of complex permeability of TEGO and Fe-Co oxide-TEGO- 40% and Fe-Co oxide-TEGO- 50% composites.

The complex permeability  $\mu$  is a measure of microwave absorption characteristic of the material arising from magnetic interactions. Fig. 6 (a &b) shows the measured  $\mu$  ' (real part) and  $\mu$  " (imaginary part) of  $\mu_r$  in the range of 8–12 GHz for the composites. As illustrated in Fig. 6 (a &b), the values of  $\mu'$  are in the range of 0.97–0.95, 1.01–0.98, and 1.01–1.02 for TEGO-10 %, Fe-Co oxide-TEGO: 40% and Fe-Co oxide-TEGO: 50% composites respectively in the frequency range of 8–12 GHz and the values of  $\mu''$  for TEGO-10%, Fe-Co oxide-TEGO: 40% and Fe-Co oxide-TEGO: 50% composites are in the range of 0.004–0.027, 0.042–0.047 and 0.059–0.087 respectively.

Fig. 7. (a &b) shows respective dielectric  $\tan \delta_{\varepsilon}$  and magnetic  $\tan \delta_{\mu}$  loss values of the composites. The TEGO-10% composite shows  $\tan \delta_{\varepsilon}$  values in the range of 0.35-0.31.  $\tan \delta_{\varepsilon}$  values for TEGO-10%, Fe-Co oxide-TEGO: 40% and Fe-Co oxide-TEGO: 50% composites are in the range of 0.27–0.27, 0.33-0.31 respectively over 8-12 GHz. The higher  $\tan \delta_{\varepsilon}$  values in TEGO is attributed to the residual defects and groups in TEGO which not only improve the impedance match characteristic but also introduce defect polarization relaxation and groups' electronic dipole relaxation, which are all in favor of electromagnetic wave penetration and absorption [41].

The magnetic  $tan \delta_u$  values of TEGO-10% composite fluctuate between 0.004-0.027, whereas, in TEGO-10%, Fe-Co oxide-TEGO: 40% and Fe-Co oxide-TEGO: 50% composites the values fluctuate between 0.042-0.048 and 0.056-0.086, respectively. This trend suggests that the EM wave absorption mechanism of the Fe-Co oxide-TEGO composites consists of both dielectric loss and magnetic loss [42]. Comparison of  $tan \delta_{\varepsilon}$  and  $tan \delta_{\mu}$ value from Fig. 7 (a & b) indicates that the reflection of the composite is mainly dependent on the dielectric loss [43]. Herein, our results clearly show that the ability of energy storage and microwave absorption the of the composite is substantially increased by combining Fe-Co oxide with TEGO under given experimental conditions.



**Fig. 7.** Dielectric (a) and magnetic (b) tangent loss of TEGO and Fe-Co oxide-TEGO- 40% and Fe-Co oxide-TEGO- 50% composites.

The return loss (RL) curves of a metal-backed single absorbing layer were calculated in the frequency range of 8–18 GHz according to the transmission line theory. It can be expressed as the following equation: [44]

RL (dB)=20 log $ (Z_{in}-1)/(Z_{in}+1) $	(A1)
$Z_{in} = (u_{e}/\epsilon_{e})^{1/2} \tan h \left[ (i2\pi fd/c) (u_{e}\epsilon_{e})^{1/2} \right]$	(A2)

where  $Z_{in}$  is the characteristic input impedance of the absorber, f is the frequency of microwaves, d is the thickness of the absorber, and c is the velocity of microwave in free space. According to Eqs. (A1) and (A2), the RL of the absorbing material is a function of six parameters:  $\varepsilon'$ ,  $\varepsilon''$ ,  $\mu'$ ,  $\mu''$ , f, d and c. For the given EM parameters, the RL of composites with different thicknesses at each frequency can be calculated according to Eqs. (A1) and (A2). In general, materials with RL > 10 dB (90% absorption) are considered as efficient microwave absorbers.

The calculated theoretical RL curves of TEGO-10 %, Fe-Co oxide-TEGO: 40% and Fe-Co oxide-TEGO: 50% composites with varying thickness as a function of frequency are shown in Fig. 8 (a-c). All the samples show a single-peaked curve. The peak value is the maximum absorption, and the maximum RL implies a better microwave absorption property. It is found that for composite samples with 2.0 mm thickness, the absorption bandwidth under -10 dB of TEGO-10% composite is 3.4 GHz (from 13.3 - 16.7 GHz) and the maximum RL can reach up to -12.0 dB at 14.8 GHz. For Fe-Co oxide-TEGO-40 wt% composite, the maximum RL is -15.0 dB at 13.5 GHz while the absorption bandwidth for RL > 10dB is 3.5GHz (from 12.0 - 15.5 GHz). Fe-Co oxide-TEGO-50 wt% composites, the absorption bandwidth can reach up to 4.0 GHz (from 9.8- 12.8 GHz) and the maximum RL is -45.0 dB at 11.5GHz. The Fe-Co oxide-TEGO composites exhibit wider operation frequency bandwidth and more absorption intensity relative to the TEGO composite. Results show that the absorption bandwidth and the maximum RL magnitude increase with the increase in Fe-Co oxide concentration. The frequency of the maximum RL is shifted towards a lower frequency band with the increase of the Fe-Co oxide weight fraction. It is quite evident from the Fig. 8 (a-c) that as the thickness of the composite samples increases, the absorption bandwidth for RL more than 10 dB decreases. TEGO-10% composite show absorption bandwidth for RL > 10 dB is 3.5, 3.4, 3.0 and 2.8 GHz with 2.0, 2.2, 2.6 and 2.8 mm thickness, respectively. Similar trend was also observed with Fe-Co oxide-TEGO composites. Absorption bandwidth for RL > 10 dB is 3.6, 3.0 and 2.8 GHz with 2.0, 2.2 and 2.6 mm thickness respectively for Fe-Co oxide-TEGO-40%. Fe-Co oxide-TEGO-50 % show absorption bandwidth for RL > 10 dB is 5.0, 4.6, 3.8, and 3.5 GHz with 1.6, 1.8, 2.0 and 2.2 mm thickness respectively. It is clear that the RL of the composite presents a regular trend at a given weight fraction of filler material in the frequency range of 8-18 GHz. Results indicate that absorption bandwidth of 3.5 GHz (8.5-12.0 GHz, X-band) and 5.0 GHz (13.0-18.0 GHz, Ku -band) can be achieved using Fe-Co oxide-TEGO composite (50 wt%) with 2.2 and 1.6 mm thickness, respectively; whereas TEGO-10% composite show absorption bandwidth of 2.8 GHz (9.0-11.8 GHz, X-band) and 3.6 GHz (13.2-16.8 GHz, Ku-band) only with 2.8 and 2.0 mm thickness respectively.

The enhanced microwave absorption performances of Fe-Co oxide-TEGO composite relative to the TEGO

composites can mainly be attributed to the greater dielectric response and improved impedance matching. Good microwave absorption by Fe-Co oxide-TEGO based composites is also attributed to the large surface area and large aspect ratio of TEGO which may improve the multiple reflections thus allowing the trap of electromagnetic radiation and the increase of propagation path inside the absorber [**45**].



Fig. 8. Calculated electromagnetic RL of (a) TEGO-10% (b) Fe-Co oxide-TEGO- 40% and (c) Fe- Co oxide-TEGO- 50% composite with different thickness.

## Conclusion

Fe- Co oxide-TEGO composite was prepared by physical mixing of Fe- Co oxide and TEGO followed by their dispersal in PU resin. EM properties of TEGO and Fe- Co oxide-TEGO composites were studied and a microwave absorption property was theoretically calculated. Result indicates enhanced microwave absorption performances of Fe-Co oxide-TEGO composite relative to the pure TEGO composites. The absorption bandwidth below -10dB increases with increase of Fe-Co oxide conc. in composite at given thickness. As the thickness of the composite increases the bandwidth for RL > 10 dB decreases. The RL of composite shows similar trend as a function of thickness at a given filler conc. in 8-18 GHz. Additionally, the microwave absorption properties can be tuned easily by varying the loading mass percentage and the layer thickness of the samples. It provides a facile method to fabricate a potential kind of excellent microwave absorbing material with light weight, strong absorption and wide absorption band-width. Our results suggest that the composite with controllable composition can be effective for microwave absorption enhancement, and may be extended to other applications.

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#### References

- Li, X.; Feng, J.; Du, Y.; Bai, J.; Fan, H.;, Zhang, H.; Peng, Y.; Lib, F.; *J. Mater. Chem. A*, **2015**, *3*, 5535.
   **DOI:** <u>10.1039/c4ta05718j</u>
- 2. Xia, T.; Zhang, C.; Oyler, A N.; Chen, B X.; *Adv Mater*, **2013**, *25*, 6905.
- DOI: 10.1002/adma.201303088
- Liu, W.J.; Che, C. R.; Chen, J. H.; Zhang, F.; Xia, F.; Wu, S. Q.; Wang, M.; *Small*, **2012**, *8*, 1214.
   **DOI:** 10.1002/smll.201102245
- Chen, Z.D.; Wang, S.G.; He, S.; Liu, J.; Guo, L.; Cao, S. M.; J. Mater. Chem., 2013, A1,5996.
   DOI: 10.1039/C3TA10664K
- Song, W. L.; Cao, M. S.; Fan, L. Z.; Lu, M. M.; Li, Y.; Wang, C. Y.; Ju, H. F.; *Carbon*, **2014**, 77,130.
   **DOI:**10.1016/j.carbon.2014.05.014
- Singh, K.; Ohlan, A.; Pham, H.V.; Balasubramaniyan, R.; Varshney, S.; Jang, J.; Hur, H. S.; Choi, M.W.; Kumar, M.; Dhawan, K.S.; Kong, S.B.; Chung, S.J.; *Nanoscale*, **2013**, *5*, 2411. **DOI:** <u>10.1039/C3NR33962A</u>
- Che, C.R.; Peng, M.L.; Duan, F. X.; Chen, Q.; Liang, L.X.; Adv. Mater., 2004, 16, 401.
- DOI: 10.1002/adma.200306460
  8. Guan, F.P.; Zhang, F.X.; Guo, J.; J. Appl. Phys. Lett. 2012, 101, 153108.
  DOI: 10.1063/1.4758931
- Li, B.Z.; Deng, D.Y.; Shen, B.; Hu, B.W.; *Mater. Sci. Eng. B*, 2009,164, 112.
- DOI: 10.1016/j.mseb.2009.08.004
  10. Kong, J.; Liu, R.J.; Wang, L.F.; Luan, Q.L.; Itoh, M.; Machida, K.; *Appl. Phys. A*, 2011, *105*, 351.
- DOI: 10.1007/s00339-011-6593-8
  11. He, Z.C.; Qiu, S.; Wang, Z. X.; Liu, R. J.; Luan, Q. L.; Liu, W.; *J Mater Chem*, 2012, 22, 22160.
  DOI: 10.1039/C2JM33068G

- Kong, J.; Wang, L.F.; Wan, Z.X.; Liu, R.J.; Itoh, M.; Machida, K., *Mater. Lett.*, **2012**, *78*, 69.
   **DOI:** 10.1016/j.matlet.2012.03.026
- Wang, L.F.; Liu, R.J.; Kong, J.; Zhang, J.Z.; Wang, Z.X.; Itoh, M.; Machida, K.; *J. Mater. Chem.*, **2011**, *21*, 4314.
   DOI: <u>10.1039/C0JM02894K</u>
- Sachdev, V.K.; Sharma, S.K.; Bhattacharya, S.; Patel, K.; Mehra, N.C.; Gupta, V.; Tandon, R.P.; *Adv. Mat. Lett.*, **2015**, *6*, 965. DOI: <u>10.5185/amlett.2015.5935</u>
- Yan, S.; Wang, L.; Wang, T.; Zhang, L.; Li,Y.; Dai, S.; Appl. Phys. A, 2016, 122, 235.
   DOI: 10.1007/s00339-016-9769-4
- Wang, L.; Jia, X.; Li, Y.; Yang, F; L. Zhang, Liu, L.; Ren, X.; Yang, H.; J. Mater. Chem., A2, 2014 14940. DOI: 10.1039/C4TA02815E
- 17. Qing, Y.C.; Zhou, W.C.; Luo, F.; Zhu, D.M.; *Carbon*, **2010**, *48*, 4074.

DOI: <u>10.1016/j.carbon.2010.07.014</u>

- Kumar, N.; Kumar, R.; Nanotechnology and Nanomaterials in the Treatment of Life-threatening Diseases; Elsevier: Netherland, 2013.
- 19. Wang, T.; Li, Y.; Wang, L.; Liu, C.; Geng, S.; Jia, X.; Yang, F.; Zhang, L.; Liu, L.; You, B.; Ren, X.; Yang, H.; *RSC Adv.*, 2015, 5, 60114.
  DOI: <u>10.1039/C5RA09715K</u>
- Wang, Z.G; Gao, Z.; Tang, W.S.; Chen, Q.C.; Duan, F.F.; Zhao, C.S.; Lin, W.S.; Feng, H.Y.; Zhou, L.; Qin, Y.; ACS Nano, 2012, 6,11009.
   DOI: 10.1021/nn304630h
- Saini, P.; Choudhary, V.; Singh, P.B.; Mathur, B.R.; Dhawan, K.S.; Synth. Met., 2011, 6, 1522.
   DOI: 10.1016/j.synthmet.2011.04.033
- Saini, P.; Arora, M.; Gupta, G.; Gupta, K. B.; Singh, N.V.; Choudhary, V.; *Nanoscale*, 2013, *5*, 4330.
   DOI: <u>10.1039/c3nr00634d</u>
- Tiwari, A.; Adv. Mat. Lett., 2012, 3, 172
   DOI: <u>10.5185/amlett.2012.2002</u>
- Chaturvedi, A.; Tiwari, A.; Tiwari, A.; Adv. Mat. Lett., 2013, 4, 656.
   DOI: 10.5185/amlett.2013.4469
- Russo, P.; Hu, A.; Compagnini, G.; *Nano-Micro Lett.*, **2013**, 5, 260.
- **DOI:** <u>10.5101/nml.v5i4</u> 26. Wang, C.; Han, X. J.; Xu, P.; Zhang, X.L.; Du, Y.C.; Hu, S.R.; Anal Phys Lett. **2011** 08 072906
- Appl. Phys. Lett., **2011**, 98, 072906. **DOI**: <u>10.1063/1.3555436</u>
- Pan, H.G.; Zhu, J.; Ma, L.S.; Sun, B. G.; Yang, J.Y.; *Appl. Mater. Interfaces*, **2013**, *5*, 12716.
   **DOI:** 10.1021/am404117v
- Zhao, C. X.; Zhang, M. Z.; Wang, Y. L.; Xi, K.; Cao, Q. Q.; Wang, H. D.; Yang, Y.; Du, W.Y.; *Sci. Rep.*, **2013**, *3*, 3421. DOI: <u>10.1038/srep03421</u>
- Sun, X.; He, P. J.; Li, X. G.; Tang, J.; Wang, T.; Guo, X.Y.; Xue, R. H.; *J. Mater. Chem.*, **2013**, *C1*, 765.
   **DOI:** <u>10.1039/C2TC00159D</u>
- Zhu, T. Z.; Sunx X.; Xue, R. H.; Guo, H.; Fan, L. X.; Pan, C. X.; He, P. J.; J. Mater. Chem., 2014, C2, 6582.
   DOI: 10.1039/C4TC00757C
- Zhang, H.; Xie, A.; Wang, C.; Wang, H.; Shen, Y.; Tian, X.; J. Mater. Chem., 2013, A1, 8547.
   DOI: 10.1039/C3TA11278K
- Kong, L.; Yi, X.; Zhang, Y.; Yuan, X.; Fang, Y.; Cheng, L.; Zhang, L.; *J. Phys. Chem. C*, **2013**, *117*, 19701.
   DOI: <u>10.1021/jp4058498</u>
- Wang, T. et al.; J. Appl. Phys., 2013, 113, 024314.
   DOI: 10.1063/1.4774243
- Gupta, V.; Patra, M. K.; Shukla, A.; Saini, L.; Songara, S.; Jani, R. K.; Vadera, S. R.; Kumar, N.; *Sci. Adv. Mater.*, **2014**, *6*, 1.
   **DOI:** 10.1166/sam.2014.1889
- 35. Hummers, S.W.; Offeman, E.R.; J. Am. Chem. Soc., 1958, 80, 1339.

**DOI:** <u>10.1021/ja01539a017</u>

 Tong, C; Advanced Materials and Design for Electromagnetic Interference Shielding; Taylor & Francis: UK, 2009. DOI: <u>10.1201/9781420073591.ch1</u>

- Savi, P.; Trinchero, D.; Tascone, R.; Orta, R.; *IEEE Trans. on Microwave Theory and Techniques*, **1997**,45, 221.
   **DOI:** 10.1109/22.557603
- Zhang, Li.; Xinxin, Y.; Hongrui, H.; Yang, L.; Mingzai, W.; Zhongzhu, W.; Guang, L.; Zhaoqi, S.; Changle, C.; *Sci. Rep.*, *5*, 9298.
   DOI: 10.1038/srep09298
- Singh, V. K.; Shukla, A.; Patra, M. K.; Saini, L.; Jani, R. K.; Vadera, S. R.; Kumar, N.; *Carbon*, **2012**, *50*, 2202.
   **DOI:** <u>10.1016/j.carbon.2012.01.033</u>
- Singh, V. K.; Patra, M. K.; Manoth, M.; Gowd, G. S., Vadera, S. R.; Kumar, N. *New Carbon Mater.*; 2009, 24, 147. DOI: <u>10.1016/S1872-5805(08)60044-X</u>
- Wang, C.; Han, X.; Xu, P.; Zhang, X.; Du, Y.; Hu, S.; Wang, J.; Wang, X.; *Appl. Phys. Lett.*; **2011**, *98*, 072906.
   DOI: <u>10.1063/1.3555436</u>
- Chen, Z. D.; Wang, S. G.; He, S.; Liu, J.; Guo, L.; Cao, S. M. J.; Mater. Chem. A., 2013, 1,5996.
   DOI: <u>10.1039/C3TA10664K</u>
- Liu, B. P.; Huang, Y.; Wang, L.; Zong, M.; Zhang, W; Mater. Lett., 2013, 107, 166.
- DOI: <u>10.1016/j.matlet.2013.05.136</u>
  44. Qin, F.; Brosseau, C.; *J. Appl. Phys.*, **2012**, *111*, 061301.
  DOI: <u>10.1063/1.3688435</u>
- Singh, P. A.; Garg, P.; Alam, F.; Singh, K.; Mathur, B.R.; Tandon, P. R.; Chandra, A.; Dhawan, K.S.; *Carbon*, **2012**, *50*, 3868.
   **DOI:** <u>10.1016/j.carbon.2012.04.030</u>