

## Preparation absorption microwave X-band based carbonyl iron/polypyrrole nanocomposite

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Received: June 2018; Revised: July 2018; July 2018

**Abstract:** The composites of “Carbonyl iron (CI)” /polypyrrole (PPy) were synthesized via different methods by in-situ polymerization on the surface of nanoparticles (NPs) with core-shell structure. This paper describes a method for polyacrylic acid (PAA) coating of NPs in aqueous solution. Then PPy coating was performed by template polymerization on NPs-PAA. The enhancement mechanism is thought due to suitable interaction and better electromagnetic match between these materials. Morphology, magnetic and conductivity properties were observed via scanning electron microscopy (SEM) and four probe method, respectively. The microwave characterization of nanocomposite was evaluated through arch test based on a network analyzer. The microwave characterization of nanocomposite was evaluated through arch test based on a network analyzer. The PPy nanocomposites possessed the excellent microwave X-band absorbers properties in 8-12 GHz. The best microwave absorption were obtained in 9.5 GHz for PPy, 8 GHz for CI-PAA-PPy with minimum reflection loss (RL) in -18.5, -18 and -17.5 dB at the thickness of 1 mm, respectively. It was also found that nanocomposites with 50% w/w and light weight exhibit good microwave absorbing properties.

**Keywords:** CI, Nanocomposites, Microwave, Polyacrylic acid, Polypyrrole.

### Introduction

In the last decade, microwave (MW) absorption materials have been widely investigated for electromagnetic interference to protect human health and electronic equipments from electromagnetic pollution which is caused by the wide applications of high-power electronic devices and communication technology [1,2]. The electromagnetic and microwave absorption properties of the carbonyl iron powders [3-5]. Nano-size manganese ferrite conductive polypyrrole composites reveal a core-shell structure by in situ polymerization [6]. For instance, Sui and et al. synthesized three-dimensional carbonyl iron powder composite which exhibit MW absorption property [7].

The conductive and magnetic nanocomposites with core-shell and different nanostructures were used for electromagnetic absorption application [8-12]. In the work, we attempted to CI nanoparticles composite to synthesize applied to prepare by in-situ polymerization on the surface of all components after were coated by PAA. This paper is a revised and expanded microwave absorption property in the frequency of 8-12 GHz.

### Results and discussion

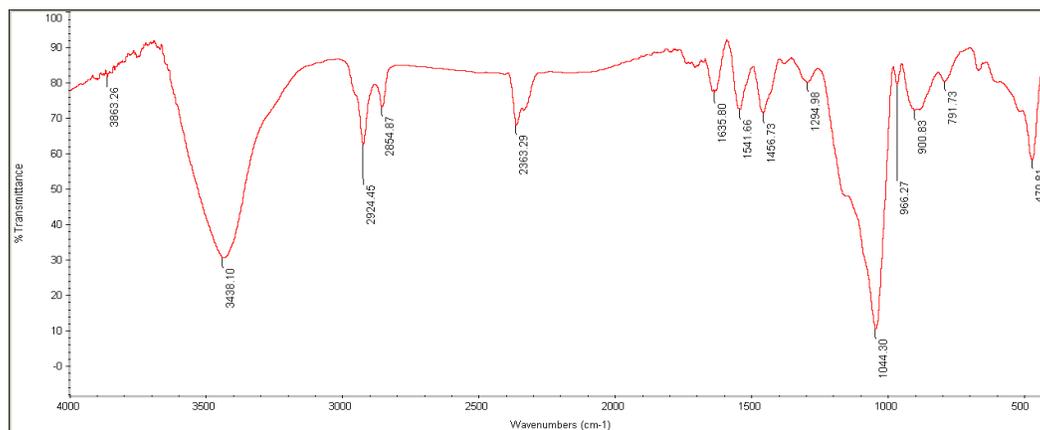
#### FTIR study:

Figure 1 shows FTIR spectrum of CI-PAA-PPy vibrations of PPy, The peak at 2924 and 2854  $\text{cm}^{-1}$  are attributed to C-H st. vibrations of PAA DBSA. The peaks at 2368  $\text{cm}^{-1}$  and 1635  $\text{cm}^{-1}$  are related to C=O st. The specific peaks around 1541 and 1481  $\text{cm}^{-1}$  are attributed with vibrational modes of quinonic and aromatic type ring for PPy. The peaks at 1169 and

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1044  $\text{cm}^{-1}$  are attributed to C-N and C-C st. vibration mode for PPy. Due to space congestion and bond angles in iron carbonyl, C = O bond does not appear in 1975, 2012, and 2032  $\text{cm}^{-1}$  [13]. Due to polypyrrole polymerization, which has a high concentration on the polypyrrole composite surface, due to space

congestion. Reducing their space congestion and also decreasing its peak intensity by coating with PAA, makes it possible to have C = O fineness week peaks in 1711  $\text{cm}^{-1}$  and in these areas. Courses under 1000 such as 473 and 585, 791  $\text{cm}^{-1}$  also relate to Fe oxidation.



**Figure 1:** FTIR spectrum of CI-PAA-PPy

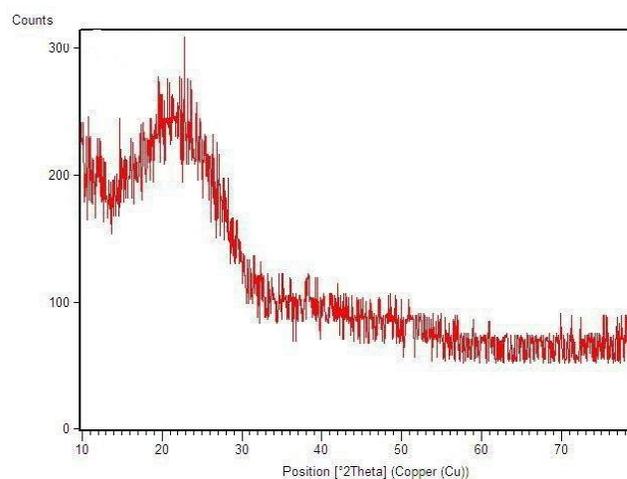
#### XRD patterns study:

Figure 2 shows XRD pattern for CI-PAA-PPy respectively. The results show the diffraction peaks of NPs structures were not destroyed after the chemical polymerization of PPy as shell. According to Figure 2 characteristic peaks at  $2\theta=16-25$  with base peaks at  $2\theta=24.83$  were observed for the CI-PAA-PPy, that correspond to JCPDS files no. 4073-004-98, respectively. These results indicated that the nanocomposite containing NPs-PAA-PPy are semi-crystalline and amorf. We can make sure of their's existence of NPs form XRD peaks The average crystallite size can be calculated by the Debye-scherrer formula:  $D = 0.89 \lambda / \beta \cos \theta$  where  $\lambda$  is the wavelength of Cu  $K_{\alpha}$  radiation and the value of K depends on serveral factors, including the Miller index of reflection plane and the shape of the crystal. If the shape is unknown, K is often assigned as a value of 0.89, D is average crystallite size,  $\theta$  is the Bragg's angle, and  $\beta$  is the full width at half-maximum of the diffraction peaks, From the obtained peak width of XRD patterns, the average crystallite sizes of CI, are calculated to 21.5, respectively.

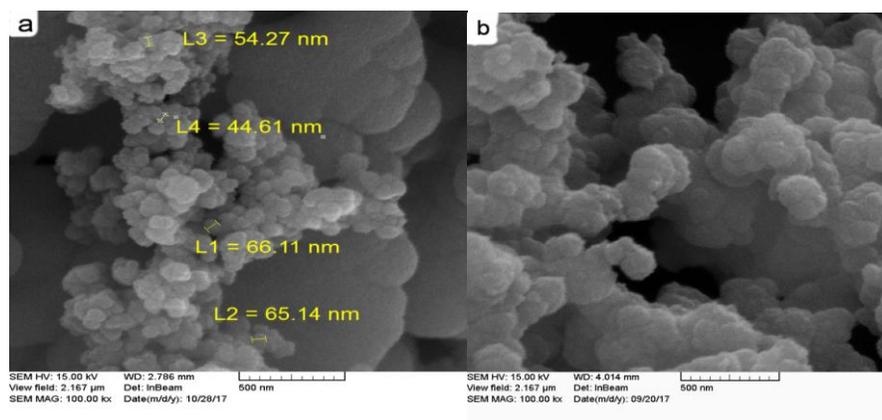
#### SEM images study:

Figure 3 (a-b) shows FESEM images of CI NPs and CI-PAA-PPy nanocomposites.

The diameters of sample are about 59 and 65 nm, respectively. All NPs are completely coated by PPy. The thickness of PPy as shell in all nanocomposites are about 10-20 nm. The surface of SEM images of nanocomposites was shown uniformity with some hollow and sponge structures.



**Figure 2:** XRD pattern for of CI-PAA-PPy



**Figure 3:** FESEM images of a) CI NPs and b) CI-PAA-PPy nano composite

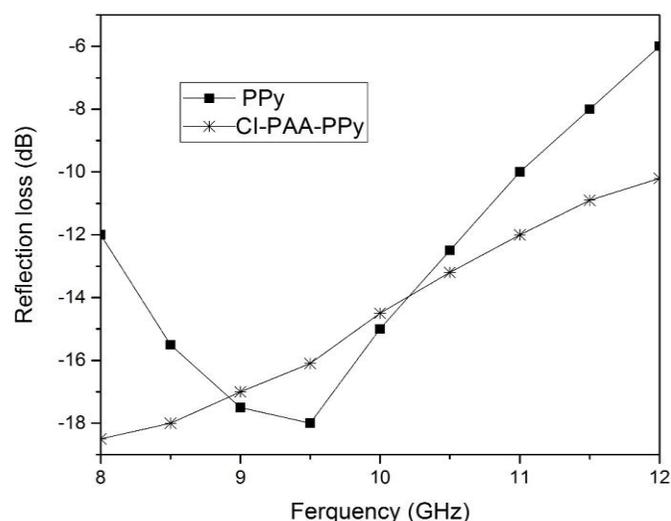
### Electrical conductivity study:

Electrical conductivity of NPs and their nanocomposites were measured by four probe methods and were summarized in table 1. The conductivity of PPy after polymerization by Fe(III) as initiator and DBSA as dopant is 0.044 S/cm. When mass content of CI as core and PPy as shell were incorporated in composites, conductivities are increased.

sample	Conductivity (S/cm)
PPy (doped)	0.044
PPy (undoped)	$1.4 \times 10^{-6}$
CI-PAA-PPy	0.083

### Microwave absorbing study:

The microwave absorbing properties of nanocomposites with the coating thickness of 1 mm investigated by using vector network analyzers in the frequency range of 8-12 GHz, that this range is contained X band. The results for PPy, CI-PAA-PPy. The best microwave absorption were obtained in 9.5 GHz for PPy, 8 GHz for CI-PAA-PPy, with minimum reflection loss in -17.5, -10, -17 and -18 dB, respectively the absorption band with under -10 dB are 9.5 and 8 GHz ranging from 8 to 12 GHz for PPy, CI-PAA-PPy.



**Figure 4:** Microwave absorbing The results for PPy, CI-PAA-PPy

### Conclusion

We have synthesized CI and described a method for PAA coating on these. Then PPy coating was performed on template polymerization via in-situ method. Finally, we prepared their nanocomposites either separately or complex with core-shell structure. In continue, their microwave absorption properties in range of 8-12 GHz, X band were investigated. The results shows that the optimum absorption are 8-9.5 GHz with RL of dB and thickness of 1 mm, respectively the microwave absorption of samples were increased by increasing core and PPy weight ratio and electrical conductivity. These samples can be used to microwave absorption as most X-band absorber for civil and military applications.

## Experimental

### Materials and Method:

Pyrrrole monomer (analytical grade, Merck) distilled twice under reduced pressure and stored below 0 °C. The liquid carbonyl iron was commercially from Aldrich. Dodecylbenzene sulfonic acid (DBSA, 90% ) and polyacrylic acid were purchased from the Aldrich.

### Coating of NPs with PAA (NPs-PAA):

0.5 g NPs and 50 ml PAA (5% w/v) were added into 250 mL flask and the mixture was ultrasonicated for 15 min. The mixture was stirred vigorously at 25 °C for 24 h. The mixture was filtered and then washed with acetic acid (2% v/v) and acetone. After vacuum drying the filtrate, NPs-PAA were achieved.

### Characterization:

The ultrasonic experiment was carried out by an ultrasonic disperser (Hielsche, UP4005, Germany). The FTIR analysis was performed using Thermo U.S. of AVATAR model. Field emission scanning electron microscopy (FESEM) were performed by TESCAN MIRA to observe surface morphologies of samples. The magnetic measurements were carried out at room temperature using a Terno company 7400 model (USA), vibrating sample magnetometer (VSM) with maximum magnetic field of 10 kOe. The XRD patterns of the samples were collected on a Philips-PW 1800 with Cu K $\alpha$  radiation ( $\lambda=1.54184 \text{ \AA}$ ) in the  $2\theta=4-90^\circ$  with steps of  $0.02^\circ$ , scanning operated at 40 kV and 30 mA (Netherlands). The electrical conductivities of compressed pellets of samples and nanocomposites were calculated using a standard four-probe set-up connected to a Keithley system comprising a voltmeter and constant high-current source, made in IRAN. Microwave absorption properties of nanocomposites were measured using microwave vector network analyzer (Agilent technologies Inc. 8722-USA) in the 2-18 GHz range at room temperature.

### Acknowledgements

Thanks to East Tehran Branch, Islamic Azad University for support and Islamshahr Branch, Islamic Azad University for cooperation.

### References

- [1] Chen, Z.; Xu, C.; Ren, W.; Cheng, H.; Adv. Mater., **2013**, *25*, 1296.  
 [2] Varshney, S.; Ohlan, A.; Iain, V. K.; Dutta, V. P.; Dhawan, S. K.; Mater. Chem. Phys., **2014**, *143*, 806.

- [3] Hongyu, W.; Dongmei, Z.; Wancheng, Z.; Fa, L.; Int J Magnetics Electromagnetism., **2016**, *2*, 005.  
 [4] Liu, L.; Duan, Y.; Ma, L.; Liu, S.; Yu, Z.; Applied Surface Science., **2010**, *3*, 842.  
 [5] Liu, L.; Duan, Y.; Liu, S.; Chen, L.; Guo, J.; J of Magnetism and Magnetic Materials., **2010**, *13*, 1736.  
 [6] Chakraborty, H., Chabri, S.; Bhowmik, N.; Transactions On Electrical And Electronic Materials., **2013**, *6*, 295.  
 [7] Sui, M.; Lu, X.; Xie, A.; Xu, W.; Rang X.; Wu, G.; Synthetic Metals., **2015**, *210*, 156.  
 [8] Hosseini, S. H.; Rahimi, R.; Kerdari, H.; Polymer Journal., **2011**, *43*, 745.  
 [9] Sedlacik, M.; Pavlinek, V.; Saha, P.; Svrčinová, P.; Filip, P.; Stejskal, J.; Smart Materials And Structures., **2010**, *19*, 115008.  
 [10] Hosseini, S.H.; Moghimi, A.; Moloudi, M.; Mater. Sci. Semicond. Proc., **2014**, *24*, 272.  
 [11] Hosseini, S.H.; Asadnia, A.; Moloudi, M.; Mater. Res. Innovations., **2015**, *19*, 107.  
 [12] Hosseini, S. H.; Sadeghi, M.; Curr. Appl. Phys., **2014**, *14*, 928.  
 [13] Moor, J. N.; Hansen, P. A.; Hochstrasser, R. M.; Biophysics., **1998**, *85*, 5062.