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# Preparation and microwave absorption properties of rod-like iron phthalocyanine with nitrile and nitro groups

Zicheng Wang, Wei Yang, Junji Wei, Fanbin Meng, Xiaobo Liu\*

Research Branch of Advanced Functional Materials, Institute of Microelectronic & Solid State Electronic, High-Temperature Resistant Polymers and Composites Key Laboratory of Sichuan Province, University of Electronic Science & Technology of China, Chengdu 610054, PR China

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## ABSTRACT

A novel iron phthalocyanine with nitro and nitrile groups (TnFePc-CN) micro-nano-structure was prepared via a simple and effective solvent-thermal route. SEM, FT-IR, UV-vis and DSC indicated that highly polar nitro and nitrile groups were successfully incorporated in phthalocyanine molecule. The strong permanent dipole moment endowed phthalocyanine with excellent microwave absorbing properties. The large microwave absorbing peak of TnFePc-CN arising at 15.8 GHz, originated from polarization relaxation of the polar nitrile groups, and achieved a maximum absorbing value of  $-9.76$  dB at the thickness of 3.7 mm.

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## 1. Introduction

As a class of porphyrin derivative, phthalocyanine (Pc) has received increasing attention due to its extensive applications, such as photocatalysis [1–3], microelectronics [4], organic field effect transistors [5], photovoltaic cell [6,7], chemical sensors [8] and light-emitting diodes [9]. Recently, a lot of novel works have been attempted in the field of microwave absorption [10,11]. However, those studies are mainly concentrated on preparation of metal-phthalocyanine and inorganic magnetic hybrid materials with special electromagnetic properties. The excellent microwave absorbing performance is primarily attributed to the introduction of inorganic magnetic material. However, the study of intrinsic microwave absorbing properties of phthalocyanine has been little reported.

Due to the formation of heterocyclic molecule with high symmetry, planarity and electron delocalization, distortion of the electron cloud around phthalocyanine ring or atoms through the presence of an external electric field can only induce a temporary and weak dipole movement, which limits their use in the field of electromagnetic absorption [12]. Hence, in order to get a high microwave absorbing intensity, introducing a strong permanent dipole moment into the Pc molecule will be a potential candidate method [13]. In this paper, we report a facile one-pot solvothermal synthesis of rod-like iron phthalocyanine with high polar nitro and nitrile groups (TnFePc-CN). The presence of nitro and nitrile

groups in TnFePc-CN endows the Pc molecule with strong polarity, which brings a comparatively strong permanent dipole movement and promotes the absorption of microwaves. Microwave electromagnetic parameters of TnFePc-CN show that the new system possesses high loss tangent of the permittivity and permeability in  $K_u$ -band. Moreover, a strong microwave absorbing peak of TnFePc-CN arises at 15.8 GHz and achieves a maximum absorbing value of  $-9.76$  dB at the thickness of 3.7 mm.

## 2. Experimental

BPh, 4,4'-bis(3,4-dicyanophenoxy)biphenyl, was synthesized in our laboratory. The synthetic procedure and characterization were reported previously [14]. 4-Nitrophthalonitrile (TnPh) (>98%) was purchased from Shijiazhuang Alpha Chemical Co. Ltd.  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  (ferrous chloride tetrahydrate), ammonium molybdate, and ethylene glycol were purchased from Kelong reagent Co. Ltd., Chengdu. All the materials were used without any further purification.

The synthesis of rod-like iron phthalocyanine was described as follows: TnPh (0.75 mmol), BPh (0.25 mmol),  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  (0.25 mmol), and ammonium molybdate (15 mg) were added into 40 ml of ethylene glycol. The mixture was stirred vigorously for 30 min and sealed in a Teflon lined stainless-steel autoclave. The autoclave was maintained at 200 °C for 15 h with ethylene glycol up to 80% of the total volume, and cooled to room temperature naturally. The obtained samples were washed with distilled water and acetone for removing residual TnPh and BPh, then dried at 50 °C for 8 h.

\* Corresponding author. Tel./fax: +86 28 83207326.

E-mail address: [liuxb@uestc.edu.cn](mailto:liuxb@uestc.edu.cn) (X. Liu).

The synthesized products were characterized by Fourier transform infrared spectrophotometer (FTIR) (Shimadzu, 8000S), scanning electron microscopy (SEM) (JSM, 6490LV). DSC analysis was carried out under  $N_2$  atmosphere at a heating rate of  $10\text{ }^\circ\text{C}/\text{min}$  using TA Q100. UV–vis absorption spectra were recorded on a UV2501-PC spectrophotometer. The electromagnetic parameters of the sample was measured on a vector network analyzer (Agilent 8720ET) at 0.5–18 GHz, in which it was mixed with wax in a mass ratio of 3:1 and compressed to standard ring shape with an outer

diameter of 7.0 mm, inner diameter of 3.0 mm, and thickness of 2–4 mm.

### 3. Results and discussion

The SEM micrographs of rod-like TnFePc-CN micro-nanostructure with different magnifications are shown in Fig. 1. As shown in Fig. 1, it is obviously observed that TnFePc-CN develops in

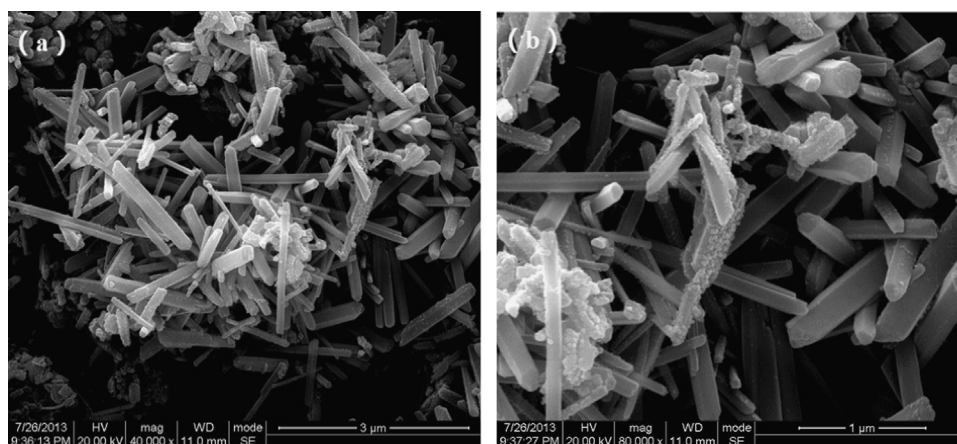


Fig. 1. (a) The SEM micrographs of TnFePc-CN micro-nanostructure and (b) high-magnification SEM image of TnFePc-CN micro-nanostructure.

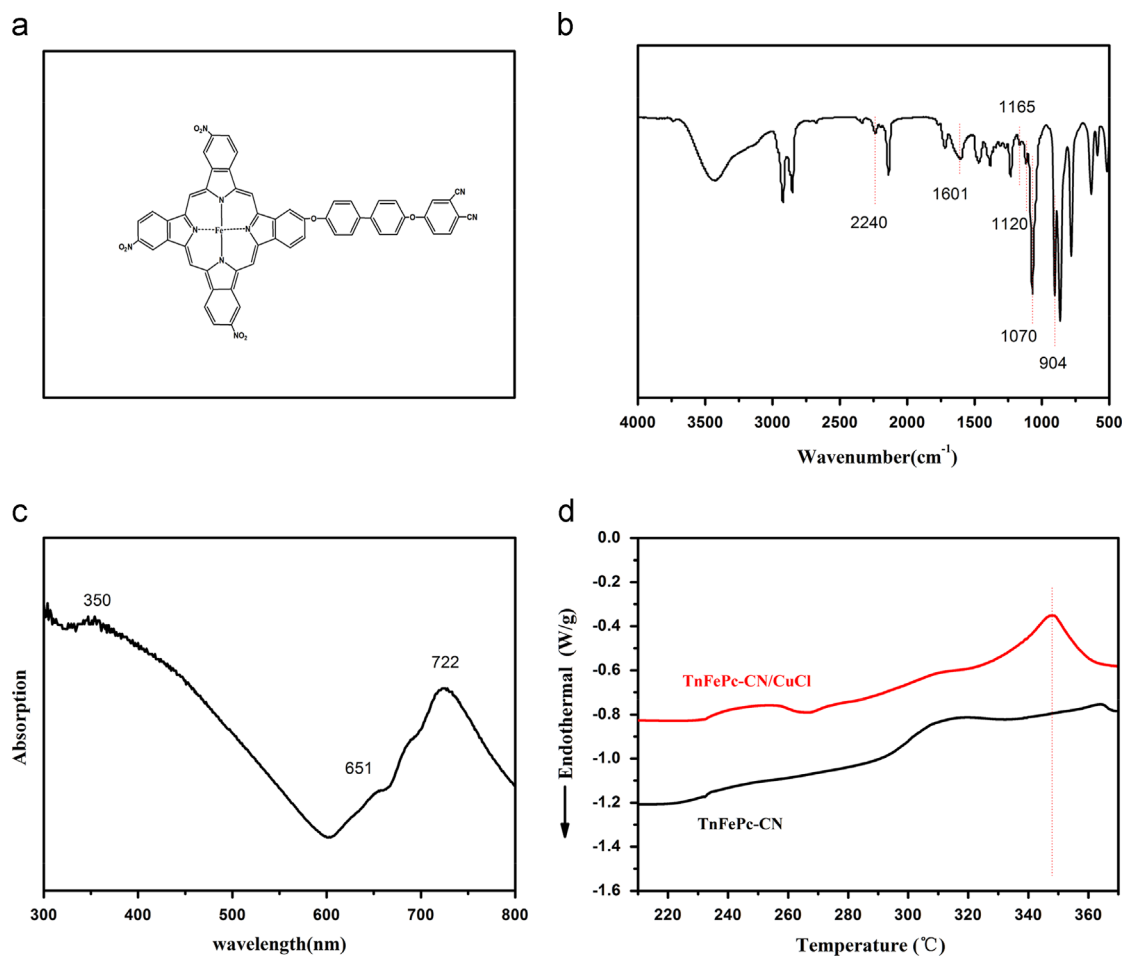


Fig. 2. (a) Molecule structure of TnFePc-CN; (b) FTIR spectrum and (c) UV–vis spectra of TnFePc-CN; (d) DSC curves of TnFePc-CN and TnFePc-CN/CuCl.

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