Preparation and Characterization of Fe_3O_4/Cellulose Composite Material by the Method of Solidoid Compound

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Abstract. The purpose of this study is to investigate the preparation and characterizations of Fe_3O_4/cellulose composite material by the method of solidoid compound. Results showed that the Fe_3O_4/cellulose composite in sheet form has been prepared by a solid reaction process. Fe_3O_4 nanoparticle was distributed on the surface of cellulose through Van der Waals force. The thermal stability of cellulose has been also improved, and the reaction ratio of cellulose was about 28.8 % ,higher than liquid homogeneous process.

Introduction

With the development of science and technology, electromagnetic pollution was more serious, harmful for health. Therefore, study on preparation of microwave absorbing materials became an issue for novel functional materials. Considering the defects of ferrite as microwave absorbing material, composite technology could combine the advantages of organics and inorganics, possesses well magnetic properties of inorganics and low density, high intensity of organics.

Solid phase doped composite is consider to be that two or more solid phase material is applied by direct composite method under certain conditions to obtain a composite material[1]. This paper studies the Solid phase doped composite method, making Fe_3O_4 nanoparticles and regenerated cellulose nano-composite direct tabletting in a solid state, in order to obtain an excellent sheet-like composite absorbing material.

Experimental

Materials. Sugarcane bagasse was collected from local plantations in Zhanjiang, China. Nanoscale cellulose was extracted from bagasse by high-pressure homogenization (HPH) in a homogeneous media of ionic liquid, 1-butyl-3-methylimidazolium chloride ([Bmim]Cl), synthesized according to a literature protocol [2]. Fe3O4 nanoparticles were synthesized by chemical coprecipitation.

Preparation of Fe_3O_4/Cellulose Composite. The experiment was carried out according to the method of Wang et al., (2015) [3].

SEM analysis. Microstructural images were obtained from scanning electron microscope (S-4800, Hitachi Limited,
Tokyo, Japan) at 1000 magnification and an accelerating voltage of 3.0 kV[4]

Thermal analysis. Thermal analysis measurements (TG) were carried out with a Synchronous Thermal Analysis (STA449C/4/G, Netzsch, Germany). Original cellulose, IL treated cellulose, and nanocellulose were heated from 50˚C to 700˚Cat a heating rate of 10˚C min−1. Under a nitrogen atmosphere with a gas flow of 20 ml·min−1[5]

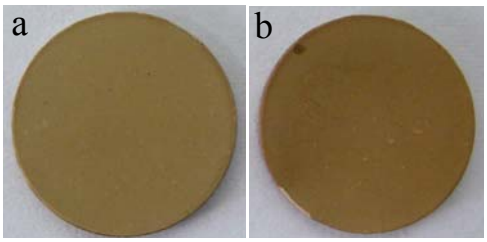
v 

Fig1.Morphologies photograph of pure cellulose flake
(a)cellulose flake without sintering, (b) cellulose flake with sintering at 200°C for 1h

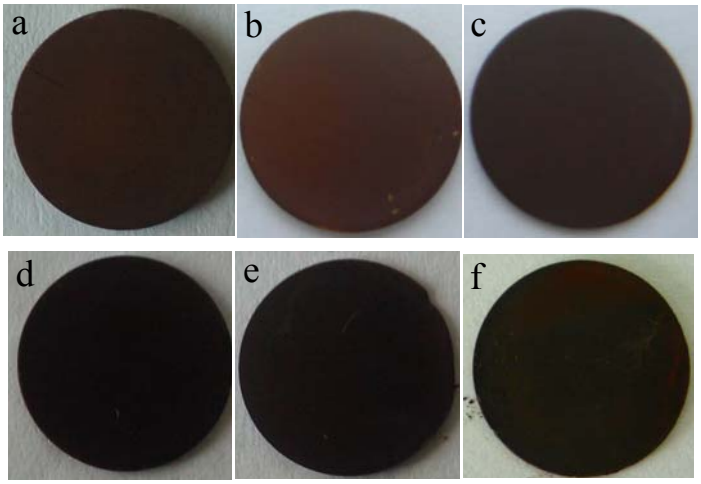


Fig 2 The morphologies photograph of solidoid compound flakes
(a), (b), (c) was the solidoid compound flakes with mass ratio of cellulose/Fe_3O_4 was 1:4, the corresponding compositing condition was: without sintering and without adding PVA; sintering at 200°C for 1h but without adding PVA; without sintering but adding 2%PVA .
(d), (e), (f) was the solidoid compound flakes with mass ratio of cellulose/Fe_3O_4 was 4:1, the corresponding compositing condition was: without sintering and without adding PVA; sintering at 200°C for 1h but without adding PVA; without sintering but adding 2%PVA .

Appearance analysis. The morphologies photograph of pure cellulose flake are shown in Fig. 1,which showed that cellulose has good molding effect under certain pressure. Fig. 1b, showed that the colour of cellulose flake with sintering at 200°C was darker. This may be attributed to that oxidation was occurred in the sintering process[6]. Fig. 2(a), (b), (c) was the solidoid compound flakes with mass ratio of cellulose/Fe_3O_4 was 1:4. Fig. 2(d), (e), (f) was the solidoid compound flakes with mass ratio of cellulose/Fe_3O_4 was 4:1. We can find that the compound of cellulose/Fe_3O_4 has good formability. This may be the reason that the appearance of Fe_3O_4 was oxidized in the sintering process.

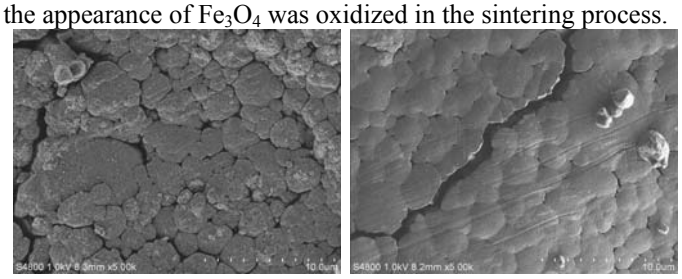


Fig. 3. SEM photograph of pure cellulose flake

The SEM photograph of pure cellulose flake are shown in Fig.3. It can be found that the surface of untreated cellulose flake (Fig 3a) arrangement closely, but there are some gap between cellulose particle. Fig 3b showed that the cellulose flake after sintering become more closely. However, a big gap was appeared on the surface. This may be that in the sintering process, the connect become more closely and the surface stress in the material increased, resulting the break of the material surface[7].

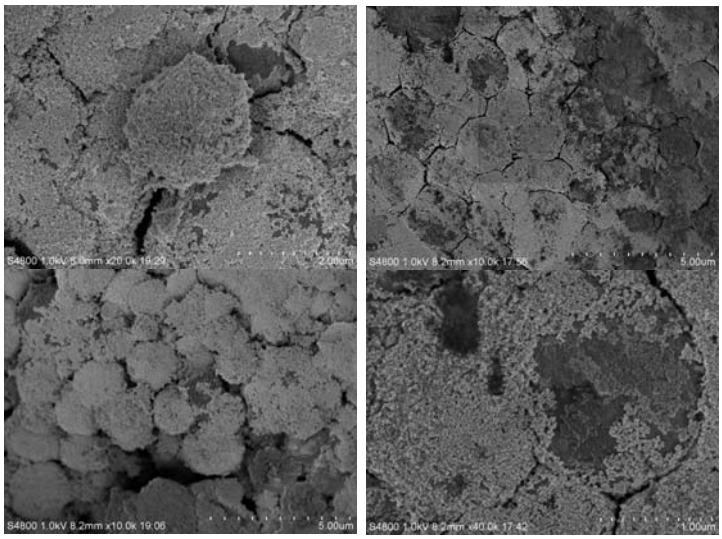


Fig. 4 SEM photograph of cellulose/Fe_3O_4 composite flake

SEM photograph of cellulose/Fe_3O_4 composite flake are shown in Fig 4. We can find that Fe_3O_4 particles was observed in the picture with cellulose effective homogeneous composite, and the smaller particle size of Fe_3O_4 particles embedded attached on the surface of the cellulose that particle size is larger. This means that the method of solid phase doping could be used to make the adsorption of Fe_3O_4 nanoparticles on the surface of nanometer fiber, resulting the formation of organic cellulose structure inorganic ,which coated with Fe_3O_4 particles.

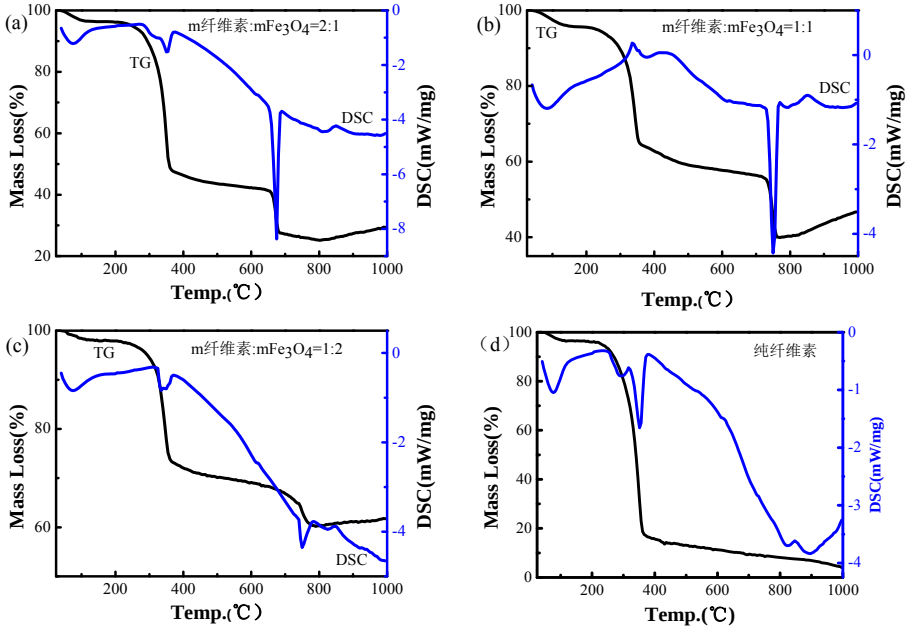


Fig.5 TG-DSC curves of Fe_3O_4/cellulose composite flake without adding adhesive

The mass ratio of cellulose/Fe_3O_4 was：（a）2:1,（b）1:1,（c）1:2,（d）pure cellulose ,

Thermostability analysis. The TG-DSC curves of Fe_3O_4/cellulose composite flake without adding adhesive are shown in Fig.5. From Fig5（a）, (b), (c)， it can be found that the curve of wight loss of samples are divided into three parts, corresponding to the three DSC endothermic paek, respectively. The temperature of the first wight loss peak was between 50°C-120°C，which was associated with the evaporation of water[8]. The accounts for about 2 %-4% of weightlessness of the total weight of the sample. The second peak of weightlessness starting from 320 °C until the end of 360 °C, which is caused by the thermal decomposition of cellulose and changed with the variation of cellulose / Fe_3O_4 mass ratio. Compared to TG -DSC curve of pure cellulose, which was shown in Figure 5 (d), a slower weight loss peak between 360 °C ~ 660 °C was appeared, which may be the reason that the the decomposition of free cellulose of the residue of cellulose. In addition, Figure 5 (a),

(b), (c) exhibited a third weight loss peak, the third weightlessness of sample in Figure 5 (a) started from 666 °C to 680 °C and reached a maximum decomposition rate at 673 °C, the weight loss of this stage was 11.7%, corresponding to a strong DSC endothermic peak.

Conclusions

The Fe_3O_4/cellulose composite in sheet form has been prepared by a solid reaction process. Fe3O4 nanoparticle was distributed on the surface of cellulose through Van der Waals force. The thermal stability of cellulose has been also improved, and the reaction ratio of cellulose was about 28.8 %, higher than liquid homogeneous process.

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