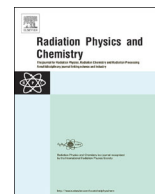




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Water dispersible polytetrafluoroethylene microparticles prepared by grafting of poly(acrylic acid)



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HIGHLIGHTS

- Preparation of water dispersible PTFE microparticles via grafting AA onto surface.
- Hydrophilicity of PTFE-g-AA was significantly improved via neutralization of NaOH.
- A very stable PTFE-g-NaAA aqueous solution can be easily prepared.

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ABSTRACT

Due to the hydrophobic nature and high gravimetric density, it is very difficult to obtain water dispersible polytetrafluoroethylene (PTFE) powder. In this work, hydrophilic PTFE microparticles were successfully prepared by grafting of poly(acrylic acid) onto PTFE micropowder via a pre-irradiation method. The as-obtained hydrophilic PTFE microparticles were analyzed by FT-IR, ¹H NMR, CA, SEM and TGA. After neutralization by sodium hydroxide, the water contact angle decreased from 145.69° for pristine PTFE to 63.38° for PTFE-g-NaAA. The obtained micropowder can be easily dispersed in water to form a dispersion with very high stability. Furthermore, the presence of grafted PAA shows no obvious influence on degradation temperature of PTFE backbones.

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1. Introduction

PTFE micropowder is usually produced by a combination of a radiation degradation technique and jet milling. Due to the excellent abrasion resistance, corrosion resistance, high thermal stability and good dielectric property, PTFE micropowder has been widely used as additives in many fields, such as inks, coatings, lubricants and composite materials (Barry et al., 2011; Anton, 1998; Liu and Zhao, 2011; Fu et al., 2004; Sparnacci et al., 2009). However, the hydrophobic nature and high gravimetric density make it very difficult to disperse PTFE micropowder in aqueous solution and limit its practical application. Although PTFE dispersion can be obtained with assistance of surfactants or via suspension polymerization, the poor stability can easily lead to precipitation of PTFE from aqueous solution. To improve the hydrophilicity of PTFE surface, several modification tactics were

developed, which usually require the use of high reactive chemicals or high energy ray inputs (Chevallier et al., 2001; Ji et al., 2002; Zhao et al., 1999; König et al., 2002; Dorschner et al., 1998; Nasef et al., 2000). Among these methods, radiation modification has been proven to be an effective approach and is widely used in many industries (Dargaville et al., 2003; Pompe et al., 2005a, 2005b). When PTFE is exposed to gamma-ray or electron beam irradiation at room temperature in air, chain scission proceeds and peroxy radicals are generated simultaneously (Schierholz et al., 1999). These radicals are very stable and can be utilized for the initiation of grafting hydrophilic monomers onto the surface of PTFE. This method has been widely applied to modify the surface of PTFE films (Turmanova et al., 1997; Adem et al., 2005) and fibers (Xiong and Yao, 2009; Wei et al., 2005) by grafting hydrophilic components. However, it is still a challenge to graft hydrophilic monomers to PTFE microparticles due to its higher hydrophobicity.

In this work, the hydrophilic PTFE microparticles were successfully obtained via grafting acrylic acid onto pre-irradiated PTFE micropowder as shown in Fig. 1. After dispersion of pre-irradiated PTFE powder in acrylic acid aqueous solution with the help of

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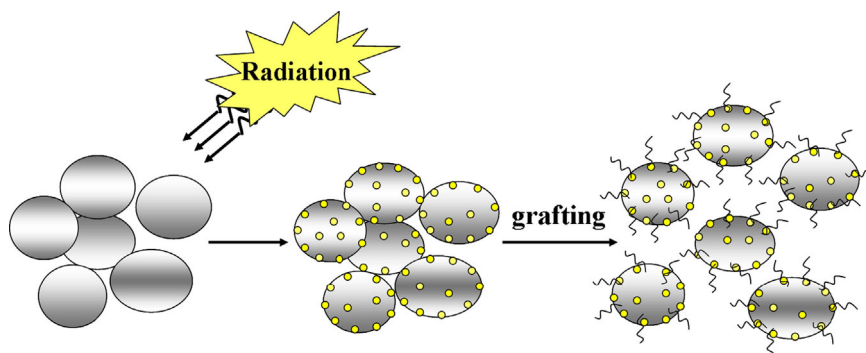


Fig. 1. Radiation induced grafting polymerization of acrylic acid onto PTFE microparticles.

surfactants, the grafting polymerization was carried out at a given temperature under a continuous stirring and a nitrogen atmosphere. Via grafting polymerization and following neutralization by sodium hydroxide, the obtained PTFE microparticles were covered by sodium polyacrylate (NaPAA), which possess extremely high hydrophilicity. With the modification of NaPAA, PTFE microparticles show significant improvement of hydrophilicity and could be easily dispersed in water to form a stable dispersion. This may indicate that these hydrophilic PTFE-g-NaAA microparticles can be applied in waterborne inks, coatings, and even lubricant.

2. Experimental section

2.1. Materials

The fluoro-containing surfactant (FSN-100) from DuPont and PTFE micropowder (diameter $\sim 5 \mu\text{m}$) from Senga Wax were used as received. Acrylic acid (AA), sodium hydroxide (NaOH) and ethyl alcohol were purchased from Sinopharm Chemical Reagent Co., Ltd. and used without further treatments.

2.2. Grafting polymerization

Prior to graft polymerization, the PTFE powder was pre-irradiated by gamma ray in air to a dose of 200 kGy for generating a large number of peroxy radicals. The content of peroxy radical in PTFE micropowder was 8.9×10^{17} spins g^{-1} , measured by an electron spin resonance (ESR) spectrometer.

For grafting experiment, 100 mL solution with different monomer concentrations and 0.05% (v/v) fluoro-containing surfactant was added into a three-neck reaction flask with a magnetic stirrer. Then 3 g pre-irradiated PTFE powder was added into solution under stirring. The reaction mixture was stirred and deaerated by bubbling nitrogen gas for 30 min, and then sealed. The obtained dispersion was heated to 65°C and stirred for a given time to complete the reaction (Chi et al., 2013; Xing et al., 2013).

After reaction, the residual monomers and homopolymers were removed by repeated centrifugation at a speed of 5000 r/min for 5 min (the obtained PTFE-g-AA microparticles were dispersed in pure water again for the following centrifugation). The degree of grafting (DG) of grafted PTFE was determined by acid-base titration with sodium hydroxide solution, according to the following equation:

$$\text{DG}(\%) = \frac{72cV}{W_0}$$

where c and V are the concentration and volume of sodium hydroxide solution, respectively, and W_0 is the weight of the PTFE powder before grafting, 72 is the molecular weight of acrylic acid.

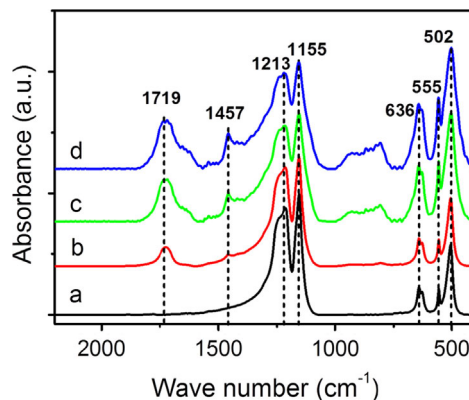


Fig. 2. FT-IR spectra of the pristine PTFE (a) and the PTFE-g-AA with DG of 21.6% (b), 26.9% (c) and 30.7% (d).

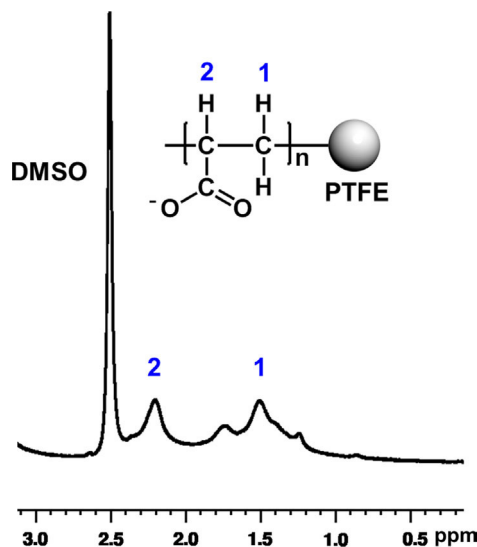


Fig. 3. ^1H NMR spectrum of PTFE-g-NaAA.

2.3. Characterization

Electron spin resonance (ESR) measurement was carried out at room temperature on a JES-FA200 ESR spectrometer using AFC balance with a frequency of 9.1 GHz at 1 mW and 100 kHz field modulation frequency. Proton nuclear magnetic resonance (^1H NMR) spectra of grafted PTFE were recorded in d^6 -DMSO solution using 500 MHz super conducting NMR spectrometer (Bruker AV 500). The PTFE-g-PAA samples were neutralized by NaOH solution before analysis. Fourier transform infrared spectra (FT-IR) were collected using a Bruker Tensor 27 FT-IR spectrometer by pressing

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