카르보닐철/ PMMA 복합물의 제조 및 자기유변학적 특성

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Preparation and characterization of polymer/carbonyl iron composite

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Introduction

Magnetorheological(MR) fluid is a stable colloidal suspension of magnetic particles dispersed in non-magnetic carrier fluid. When exposed to a magnetic field magnetic fluids transformed from a fluid-like to a solid-like state within milliseconds[1-4]. Magnetic particles building up chain-like structure with a magnetic field was due to dipole-dipole interaction. So the formed chain structure causes an increase in appearance shear stress and viscosity. MR fluid has a high yield stress up to 10-100 kPa under a strong magnetic field[5]. Controlling the intensity of the magnetic field applied in the MR fluid can adjust the apparent viscosity and other rheological properties of the fluid. These fluids can be widely used in designing damping devices such as shock absorbers, clutch and a vibration dampers[6]. The carrier phase of MR fluids can be silicone oil, mineral oil, synthetic hydrocarbon compound, water, or other suitable organic liquid. Ferromagnetic and Ferrimagnetic particles[7] were always used as magnetorheological particles since these particles are easily magnetized under magnetic field. In addition, Iron, iron oxide, iron nitrate and carbonyl iron are also used as MR particles. Among various MR materials, MR fluid based on carbonyl iron has attracted much attention to its strong magnetic properties. However, most magnetic fluids including MR suspension have sedimentation and aggregation problems due to their high density and the remnant magnetization. Therefore, the dispersion stability and the re-dispersibility of MR fluids need to be improved for more effects of MR fluids. In order to improve the stability of the MR fluids, magnetic particles were treated with polymer or surfactant on the surface[8-11]. In this study to overcome these problems, we prepared magnetic composite particles with Carbonyl iron and Poly(methyl methacrylate). The stability of this MR fluid were studied and the rheological behavior of the MR fluid in the steady shear mode and oscillation mode was also investigated.

Experimental

The micro-sized polymer spheres contained magnetic particles were synthesized by suspension polymerization. Methymethacrylate (MMA), Ethylene glycol dimethyacrylate(EGDMA) and carbonyl iron were uesd without any further purification. Poly(vinyl alcohol) was used as stabilizer. 2,2'-Azobis(isobutyronitrile)(AIBN) were used

initiator in the polymerization, after recrystrallizde from methanol. For the aqueous phase, PVA was dissolved in deionized water with constant stirring in double jacket reactor. For the oil phase, carbonyl iron particles were mixed with MMA, EGDMA and AIBN. The magnetic particle mixture was added to the aqueous phase and the solution temperature was increase to 65℃. Suspension polymerization was kept for 16h with stirring. the final PMMA-CI spheres were filtered and washed with di-water several times. Then the magnetic microspheres were dispersed in mineral oil preparing for magnetorheological fluid

The diameter and surface features of the magnetic polymer microspheres were obtained via scanning electron microscope (SEM, JSM-6700F, JEOL, Tokyo, Japan) and transmission electron micrograph(TEM). The thermogravimetric analysis (TGA) was carried out from 30 ℃ to 800℃ with a heating rate of 20℃/min under nitrogen atmosphere. The magnetite content in the magnetic polymer microspheres was calculated from residues. The crystal structure of the magnetic particles was investigated by a powder X-ray diffraction experiments conducted with a Rigaku (Copper radiation, 40kV, 100mA) in the range of 2Θ =10-80°. Magnetic properties of the magnetic polymer microspheres were measured by the vibrating sample magnetometer (VSM) at room temperature. In addition, Rheological properties of monodispersed magnetorheological suspensions of the magnetic microspheres dispersed in silicone oil were examined under a magnetic field using a parallel plate typed rheometer.

Results and discussion

Magnetic PMMA-CI microparticles were prepared by the suspension polymerzation. the morphology of polymeric magnetic spheres was observed by scanning electron microscope (SEM) image (Fig.1). the obtained particles showed spherical in shape and the average size of droplet was about 10㎛. a rough spherical structure could be observed form the surface of polymeric magnetic particles.

The transmission electron micrograph(TEM) image showed carbonyl iron located in side of the polymer sphere. The sample particles size was about 10㎛ and carbonyl iron size was about 1㎛. Fig.1 (b) showed TGA thermograms for PMMA particle and magnetic polymer spheres. a the composite shoed a 70wt% weight loss from 250℃ to 450℃, lefting a 30wt% residual which was attributed to the organic particles.

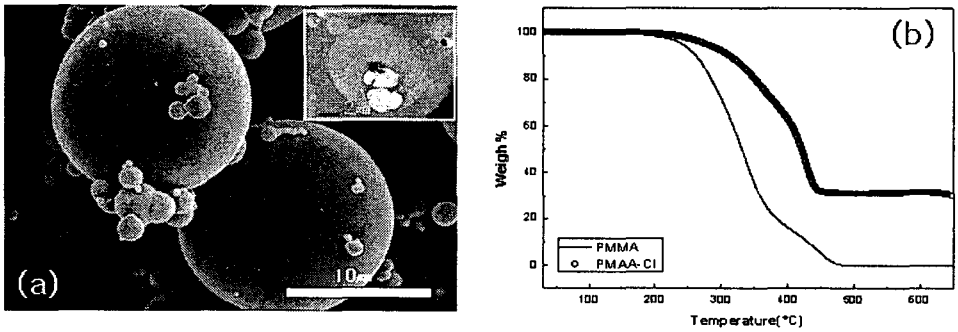

Figure 1. (a) SEM image of PMMA-CI particles and inset is the TEM image of PMMA-CI and (b) TGA thermogram of PMMA-CI and PMMA

Fig.2(a) showde XRD patterns for magnetic polymer microspehres and pure cabonyl

iron. The XRD pattern of magnetic polymeric spheres showed distinct peaks at 2θ value of 44.84 and 65.21, which correspond to characteristic peak of carbonyl iron BCC structure. This result agreed that these polymeric particles contained carbonyl iron particles.

Magnetic characterization carbonyl iron particles and magnetic polymer particles were tested by vibrating sample magnetometer(VSM)(Fig.2(b)). The saturation magnetization was found to be 72.85emu/g, and was lower than pure carbonyl iron, due to the structural disparity. that was to say, the magnetization can only be attained at the magnetic particle amount.

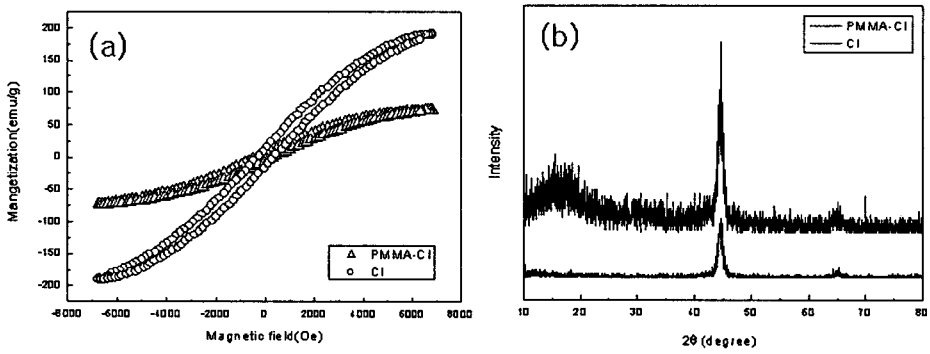

Figure 2. (a) The VSM of PMMA-CI particles and CI and (b) The XRD dirractogram of PMMA-CI particles and CI

Fig.3 (a) show shear stress and viscosity as a function of shear rate for PMMA-CI test under various magnetic field strength. At without magnetic field, and MR fluids showed a newtonian fluid like behavior. The yield stress increased by exhibiting obvious MR effect. MR fluids showed increased shear stress when applied magnetic field increased. An increase in the magnetic field strength leads to an almost exponential increase in the apparent yield point. Magnetorheological fluid change in flow property transport from a liquid-like to a solid-like state under magnetic field strength.

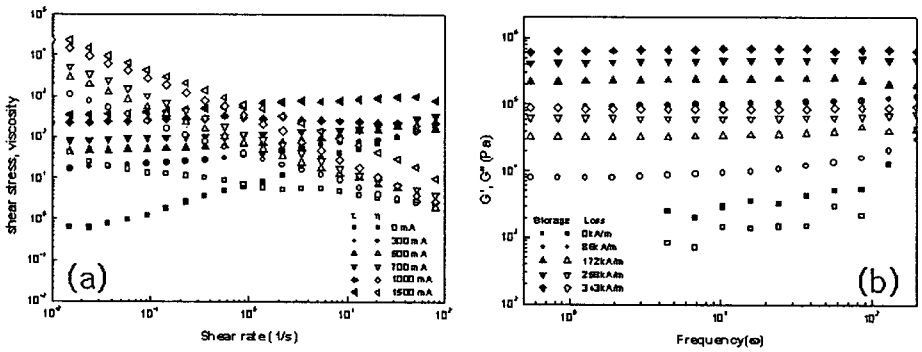

Figure 3. rheological properties of PMMA-CI particles (a) shear stress and viscosity versus shear rate (b) frequency sweep test

Magnetic particles building up chain-like structure with an applied magnetic field was due to dipole-dipole interaction. So the formed chain structure caused an increasing viscosity[12-14].

Fig.3.(b) show storage modulus and loss modulus as a function of frequency for the PMMA-CI under magnetic field strength. these results suggest that, both G' and G" responses get enhanced with increased magnetic field strength. These frequency dependence of dynamic moduli profiles suggests that under sample are more elastic (solid-like) in magnetic field, that they undergo deformation during frequency sweep[15].

Conclusions

The magnetic polymer microspheres were synthesized by suspnesion polymerization. the magnetic particles are spherical in shape. the particles were found to embed insied of the polymer spheres. The magnetic propertied decreased than that of the pure carbonyl iron because of the existence on polymer. MR suspensions of magnetic composite particles in dispersed mineral oil were prepared and their MR properties under magnetic fields were measured. The shear stress of the MR fluid was increased when increasing magnetic field. the magnetic density was increased, the yield stress got the MR fluid was also increased, exhibiting obvious MR effect. When the magnetic field strength increased, the loss modulus and storage modulus value were also increased indicating a very strong elastic properties.

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