Preparation of high toughness PAM-Gel/CNTs-RGO hydrogel and its electromagnetic shielding properties

1 Experiment

1.1 Materials

Acrylamide (AM, AR), ammonium persulfate (APS, AR), N, N' -methylene bisacrylamide (MBI, AR), gelatin (Gel), polyvinylpyrrolidone K30 (PVP, GR) purchased from Shanghai Aladdin Biochemical Technology Co., LTD.; Multi-wall carbon nanotubes (MWCNTs) and reduced graphene oxide (RGO) were purchased from Suzhou Carbon Feng Graphene Technology Co., LTD. No additional purification was performed on all reagents, and deionized water was used for solution preparation.

1.2 Preparation of PAM-Gel, PAM-Gel/CNTs, PAM-Gel/RGO, PAM-Gel/CNTs-RGO hydrogels

PAM-Gel hydrogel was prepared by hydrothermal method. Firstly, 0.2 g Gel was heated and dissolved in 10 ml deionized water. After cooling to room temperature, 2 g AM, 0.065 g APS and 0.02 g MBI were dissolved in the above solution to form a uniform mixture. After reacting at 60℃ for 4 h, PAM-Gel hydrogel was prepared by thermal initiated polymerization method. On the basis of the above, PAM-Gel/CNTs hydrogel was formed by adding different amounts of CNTs (0.05 g, 0.1 g, 0.15 g, 0.2 g) and PAM-Gel under magnetic agitation to form a uniform mixture, which was reacted at 60℃ for 5 h, and PAM-Gel/CNTs hydrogel was prepared by heat-initiated polymerization method. PAM-Gel/RGO hydrogels were prepared in the same way as PAM-Gel-CNTs hydrogels. PAM-Gel/CNTs-RGO hydrogel first dissolved CNTs and RGO in deionized water to form a uniform mixed solution, and dissolved RGO was dispersed by adding a certain amount of PVP to assist RGO, and then prepared a PAM-Gel mixed solution and poured into CNTs and RGO solution to form a uniform mixed solution by magnetic stirring. After reaction at 60℃ for 5 h, PAM-Gel/CNTs-RGO hydrogel was prepared by thermal initiation polymerization.

1.3 Properties of hydrogels

The morphology of hydrogels was observed by SEM (Quanta FEG 250, FEI Company). The FTIR spectra were recorded with ATR using the Nicolet iS50 spectrometer (Thermo, Fisher Scientific) in the range 600 to 3600 cm^{-1}. XRD (Rigaku

Ultima IV, Japan) is tested using copper targets, measuring angles of 5-90° and scanning rates of 10°/min. X-ray photoelectron spectroscopy (XPS) analysis of Thermo Scientific K-Alpha was performed with Al-Ka X-rays.

Water content (SR) can be obtained by formula (1) [1, 2]

$$SR（%）=\frac{Ws-Wd}{Ws}\times 100\% \quad （1）$$

Where Ws is the weight of the original sample and Wd is the weight of the final dried sample after drying at 70℃ for 24 h in the vacuum drying chamber. It should be noted that the sample after the moisture content test will be directly tested for swelling properties.

The dried sample (Wd) was placed in deionized water and weighed (Ws) with filter paper every hour to remove surface moisture. The measurements were repeated three times for each sample and averaged. The swelling rate (S) of hydrogel is calculated by equation (2) [3]:

$$S（%）=\frac{Ws-Wd}{Wd\times 100}\% \quad （2）$$

Mechanical performance tests (compression) were carried out on a general mechanical test platform (CMT 6104, Systems (China) Co.) at temperatures of 10-20 ° C and relative humidity of 45-50%. The mechanical test standards of hydrogel samples shall be uniformly implemented according to the national standard (GB/T 1040.3-2006). The size of the sample is cylindrical.

The sample was cut into rectangular strips of 10 mm, 5 mm wide and 4 mm thick, using a four-probe tester (RK-YA, China) : a commonly used electrical conductivity measurement equipment, according to the national standard, operating at 5 v, the temperature is 10-13℃, the relative humidity is 45-50%, and the spacing between each contact is 2.35 mm; Test at room temperature, at least three times for each sample. Voltammetry was used to determine the volume resistivity (σ) and electrical conductivity (ρ) of low conductivity samples (< 10^{-6} s m^{-1}). The calculation formula is as follows [4, 5]：

$$\sigma=\frac{1}{\rho} \quad （3）$$

The EMI test instrument used was a vector Network analyzer (N5225B, Keysight, China), and the electromagnetic interference shielding effectiveness (EMI SE) was estimated in the X-band (8.2-12.4 GHz) range. The test sample size is 10 mm long, 5

mm wide and 4 mm thick, scattering parameters S11 and S21 are obtained, and then the power coefficients of reflectance (R), transmittance (T) and absorptivity (a) are calculated using formulas (4) - (9) [6, 7], As well as reflected EMI SE (SE_R), total EMI SE (SE_T) and absorbed EMI SE (SE_A). Then vector network analyzer (N5225B, Keysight, China) was used to measure the electromagnetic parameters of the sample, namely the real and imaginary parts of the dielectric constant, by waveguide method.

$$R = |S_{11}|^2 \quad (4)$$

$$T = |S_{21}|^2 \quad (5)$$

$$A = 1 - (T + R) \quad (6)$$

$$SE_T = 10\log\frac{1}{T} \quad (7)$$

$$SE_R = 10\log\frac{1}{(1 - R)} \quad (8)$$

$$SE_A = 10\log\frac{(1 - R)}{T} \quad (9)$$

Reference

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