Synthesis, characterization and fluorescent performance studies of novel diphenyl sulfone-functionalized water-soluble polymer

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Abstract: In this communication, a novel water-soluble diphenyl sulfone-functionalized polymer was successfully synthesized through a facile hydrothermal synthesis route and then characterized by FT-IR, UV–Vis, ^{1}H-NMR and ^{13}C-NMR spectroscopy. Fluorescence quenching experiments revealed that the fluorescence intensities of the resulting diphenyl sulfone-functionalized polymers were linear with the concentrations of Fe^{3+} and 4-nitrophenol (4-NP) in the concentration ranges of $(5.0–24.9)\times10^{-8}$ and $(5.0–50.0)\times10^{-7}$ mol dm^{-3}, with detection limits of 2.8×10^{-8} and 2.2×10^{-7} mol dm^{-3}, respectively. These results created opportunities for the development of novel chemosensors by introducing selective fluorescent groups into polymeric materials.

Keywords: fluorescence sensing; 4-nitrophenol; Fe^{3+}; aqueous solution.

INTRODUCTION

With the rapid development of modern industry, a large amount of industrial wastewater is being released untreated into the natural environment, thus causing increasingly serious environmental pollution problems.[1] Among these existed pollutants, the most dangerous and toxic species are nitroaromatic compounds (NACs) and heavy metal ions, because they can cause serious and irreversible damage to the environment even at relatively low levels.[2,3] Therefore, accurate monitoring of such highly hazardous pollutants, especially at very low concentrations, is particularly important for protecting the safety of ecological environments. The commonly used techniques for the detection of pollutants are electrochemical, phosphomimetic and colorimetric methods.[4–6] However, their wide application is significantly limited due to insurmountable defects, such as complexity, high cost and relatively low accuracy. Fluorescence analysis is by far superior to the common methods due to its simplicity, sensitivity and efficiency.[7–10]

Unlike small molecules, polymer as fluorescent chemosensors have some distinct advantages, such as high sensitivity and ease of fabrication of devices.[11] To date, many efforts were primarily focused on the design and construction of polymers as highly selective and sensitive chemo/bio fluorescent sensors to detect specific analytes.[12] However, poor water-solubility of this kind of compounds restrict their application in the practical field since many pollutants exist in an aqueous environment.[13] Therefore, the study of water-soluble polymers with fluorescent groups for chemo/bio analyses is a significant research field for the benefit of economy, environmental protection and social development.[14]

Sulfur is an abundant active element that can form various compounds. These compounds have shown satisfactory performance in environmental fields owing to their thermal stability, mechanical strength, chemical inertness, high fluorescent, *etc*.[15–19] Moreover, the lone pair of electrons on the sulfur atom allows for the complexing of these compounds with different metal ions.[20] Experiments have proved that modification of polyethylenimine (PEI) with ligands usually containing sulfur donors have led to the formation of soluble polymers for binding toxic metal ions.[21] Poly(*N*-isopropylacrylamide) (PNIPAM) and polyacrylamide (PAM) have been widely applied for this purpose.[22] PNIPAM and PAM are highly hydrophilic and thus can enhance the water-solubility of hydrophobic materials.[23]

In this paper, a simple water-soluble polymer was synthesized and further applied for the selective fluorescence detection of Fe^{3+} and 4-NP in aqueous solution. The influences of different NACs and metal cations on the fluorescence intensities of the polymer were also investigated.

EXPERIMENTAL

Materials

4,4'-Dihydroxydiphenyl sulfone (DHDPS), triethylamine, methacryloyl chloride (MAC) and *N*-isopropyl acrylamide (NIPAM) were purchased from Aladdin Reagent Co. Ltd. (Shanghai, China), the remaining reagents were purchased from Tianjin Chemistry Reagent Company in China. All reagents used in the experiments were of analytical grade. Azobisisobutyronitrile (AIBN) and NIPAM were purified by recrystallization from ethanol and hexane, respectively. Deionized water (18 MΩ cm) from a water purification system was used in the experiments.

Preparation of the fluorescent copolymer

The monomer 4,4'-bis(methylacryloxy) diphenyl sulfone (BMPS) is presented in Scheme 1. A mixture containing 2.5 g (10 mmol) of DHDPS, 5.0 cm³ of triethylamine and 35.0 cm³ of tetrahydrofuran (THF) was introduced into a three-neck flask placed in an ice-bath. Then 3.0 cm³ (30 mmol) of MAC was added dropwise to the reaction mixture. Afterward, the mixture was stirred for additional 12 h at room temperature. After completion of the reaction, the solution was diluted with a large amount of distilled water. The formed precipitate was collected after several washings with distilled water. Further purification was accomplished by

recrystallization from ethanol. The obtained BMPS was dried in a vacuum oven at 50 °C (yield: 94 %).
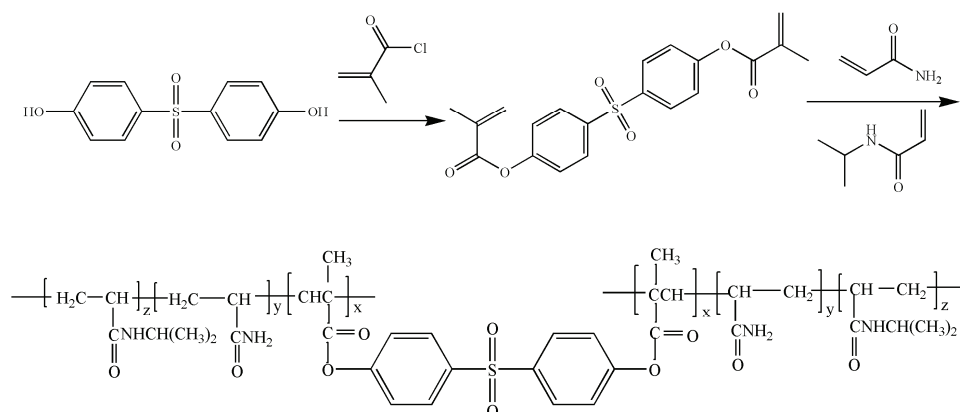
Scheme 1. Schematic illustration of the preparation of the fluorescent copolymer.

A mixture of 1.7 g (15 mmol) of NIPAM, 0.1 g (0.35 mmol) of BMPS and 2.5 g (35 mmol) of AM in THF (40 cm^3) was introduced into a three-neck flask, then initiator AIBN (0.04 g) was dissolved in 5 cm^3 THF and added into this flask. The mixture heated to at 65 °C for 24 h under a nitrogen atmosphere. After completion of the reaction, the reaction mixture was allowed to cool to room temperature, then the solution was precipitated in deionized water. After filtration, the product was dissolved in THF and reprecipitated in diethyl ether (this procedure was repeated 2 times). Finally, the precipitate was filtered and then dried to give poly(NIPAM–AM–BMPS) (yield: 82 %).

Characterization

Fourier transform infrared spectroscopy (FT-IR) measurements were performed on a Bruker Vector-22 FT-IR spectrometer. Each vacuum-dried sample was ground with KBr and compressed into a pellet.

UV–Vis spectra were taken on a Lambda UV2550 spectrometer (Perkin Elmer) and recorded in methanol/water (1:1 volume ratio) solution.

^{1}H-NMR and ^{13}C-NMR spectra were recorded on an Agilent Technologies NMR system 400 and a Bruker Avance III HD 600 MHz NMR spectrometer. The spectra were recorded in deuterated water (D$_2$O) using tetramethylsilane (TMS) as the internal standard.

The fluorescence experiments were performed at room temperature with the major equipment being a Perkin–Elmer F-4600 luminescence spectrometer (Stock solutions of 0.1 kg m^{-3} polymer probe were prepared in deionized water).

RESULTS AND DISCUSSION

FT-IR, UV-Vis and NMR analyses

The polymer was characterized by FT-IR and UV spectra (Fig. 1), The FT-IR spectrum in Fig. 1a exhibits a broad absorption peak at 3422 cm^{-1}, corresponding to the stretching vibration of N–H groups, whereas the low-intensity peak around 2968 cm^{-1} is due to the stretching vibration of sp^3 C–H,[24] the peak

at 1666 cm^{-1} is attributed to the C=O stretching vibrations (amide I band). Moreover, the peak at 1548 cm^{-1} corresponds to the *N*-monosubstituted amides (N–H bonding vibrations, amide II), the peak at 1456 cm^{-1} results from the scissoring vibrations of methylene group (CH$_2$),[25] the peak at 1343 cm^{-1} is attributed to the –CH(CH$_3$)$_2$ groups.[26] These results confirm the presence of NIPAM and AM units. Since the content of BMPS in the copolymer is low, the FTIR results cannot provide clear evidence for the existence of BMPS in the copolymer.
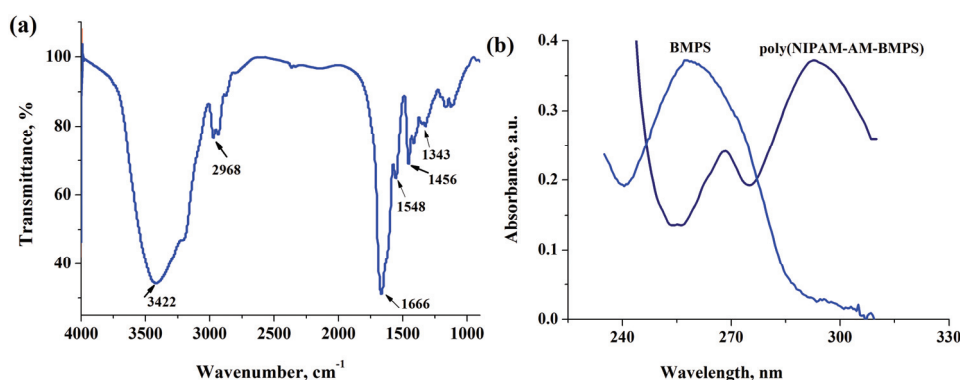

Fig. 1. a) FT-IR spectrum of poly(NIPAM–AM–BMPS), b) UV–Vis spectrum of BMPS and poly(NIPAM–AM–BMPS).

Furthermore, poly(NIPAM–AM–BMPS) was also studied by UV–Vis spectroscopy (Fig. 1b). The absorption of poly(NIPAM–AM–BMPS) is derived from the chromophore moieties of BMPS and the polymerization does not change the optical character of BMPS. Therefore, it can be concluded that BMPS participates in the polymerization of NIPAM and AM.

The structure of poly(NIPAM–AM–BMPS) was further confirmed by NMR. The ^{1}H-NMR and ^{13}C-NMR spectra are shown in Fig. 2.

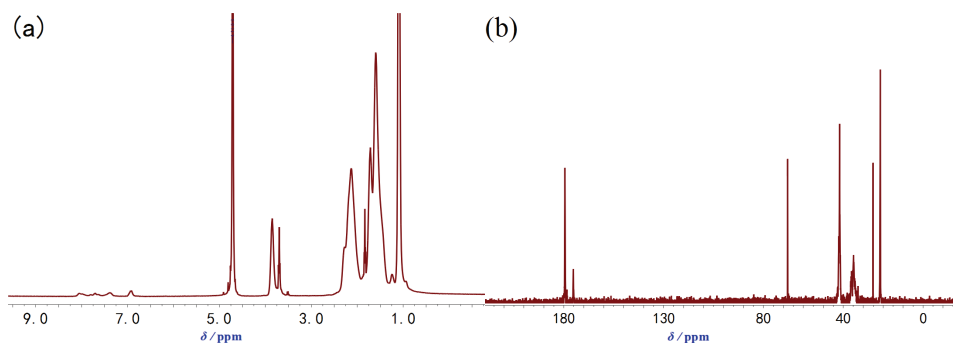Fig. 2. ^{1}H-NMR (a) and ^{13}C-NMR spectra (b) of poly(NIPAM–AM–BMPS) in D$_2$O.

^{1}H-NMR (400 MHz, D$_2$O, δ / ppm): 1.1 (CH$_3$ protons of *N*-isopropyl), 1.36–1.80 (CH$_2$ protons of the polymer backbone), 1.92–2.36 (CH protons of the polymer backbone), 3.85 (CH protons of *N*-isopropyl), 1.83 and 3.64 (residues of THF), 4.71 (protons of the deuterated solvent), 6.8–8.2 (aromatic protons).

^{13}C-NMR (101 MHz, D$_2$O, δ / ppm): 175.3 and 175.1 (C=O), 67.86 (CH), 41.84 (CHR$_2$), 35.95–34.75 (CH$_2$), 25.05 and 21.44 (CH$_3$).

Fluorescence detection of 4-NP

Poly(NIPAM–AM–BMPS) has high solubility and excellent fluorescence intensity in water. Therefore, water was selected as the solvent to explore the potential fluorescence sensing performances for the detection of 4-NP. Trinitrotoluene (TNT), 4-nitrotoluene (4-NT), 2,6-dinitrotoluene (2,6-DNT), 2,4-dinitrotoluene (2,4-DNT), 4-nitrophenol (4-NP), 2-nitrophenol (2-NP), nitrobenzene (NB), 2-nitrotoluene (2-NT) and 2,4,6-trinitrophenol (TNP) were used to evaluate the quenching efficiency of the sensing of NACs (0.1 mol m^{-3}; λ_{ex} = 250 nm, λ_{em} = 297 nm). It was found that TNT, 4-NT, 2,6-DNT, 2,4-DNT, 2-NT, 4-NP, 2-NP, TNP and NB have lower quenching efficiency for the fluorescence intensities of the probing solution, while 4-NP has an excellent quenching efficiency (Fig. 3a).

These results indicate that poly(NIPAM–AM–BMPS) possesses high selectivity for 4-NP sensing compared to the other NACs. This was the inspiration to systematically establish the applicability of sensors for the detection of 4-NP.

To better understand the sensitivity of poly(NIPAM–AM–BMPS) toward 4-NP, fluorescence quenching titration studies were performed by progressive addition of 4-NP (0.1 mol m^{-3}). On incremental addition of 4-NP, high sensitivity of sequential fluorescence quenching was clearly observed (Fig. 3b). The quenching percentage was estimated using the formula $100(I_0 - I)/I_0$ %, where I_0 and I are the fluorescence intensities of probe solution at 293 nm before and after the addition of 4-NP. The luminescence efficiency of the solution was rapidly reduced on addition of 4-NP (Fig. 3c). The fluorescence response of probing solution can easily be detected at very low concentrations of 4-NP, suggesting that the small amount of 4-NP can quench the luminescence intensity of poly(NIPAM–AM–BMPS). As shown in Fig. 3d, the sensitivity was evaluated through the Sterne–Volmer equation $I_0/I = K_{SV}[Q] + 1$, where I_0 and I are the fluorescence intensities before and after addition of 4-NP, $[Q]$ and K_{SV} are the concentration of 4-NP and the Sterne–Volmer constant, respectively. Notably, the ratio of fluorescence intensity in the absence and presence of 4-NP displays a linear response to the 4-NP concentration in the range from 5.0×10^{-8} to 24.9×10^{-8} mol dm^{-3}, and an obvious linear relationship (R^2 = 0.996) was attained. The detection limit was calculated to be 2.8×10^{-8} mol dm^{-3} ($3\sigma/k$, σ: standard deviation, n = 3) and the corresponding constant K_{SV} for 4-NP probing was calcul-

ated to be 1.46×10^6 dm^3 mol^{-1}. These results show that poly(NIPAM–AM––BMPS) has high sensitivity and can selectively detect 4-NP in aqueous solutions. To the best of our knowledge, the Ksv value obtained in this work is larger than Ksv of the sensors previously applied to detect 4-NP (Table I).[27–32]
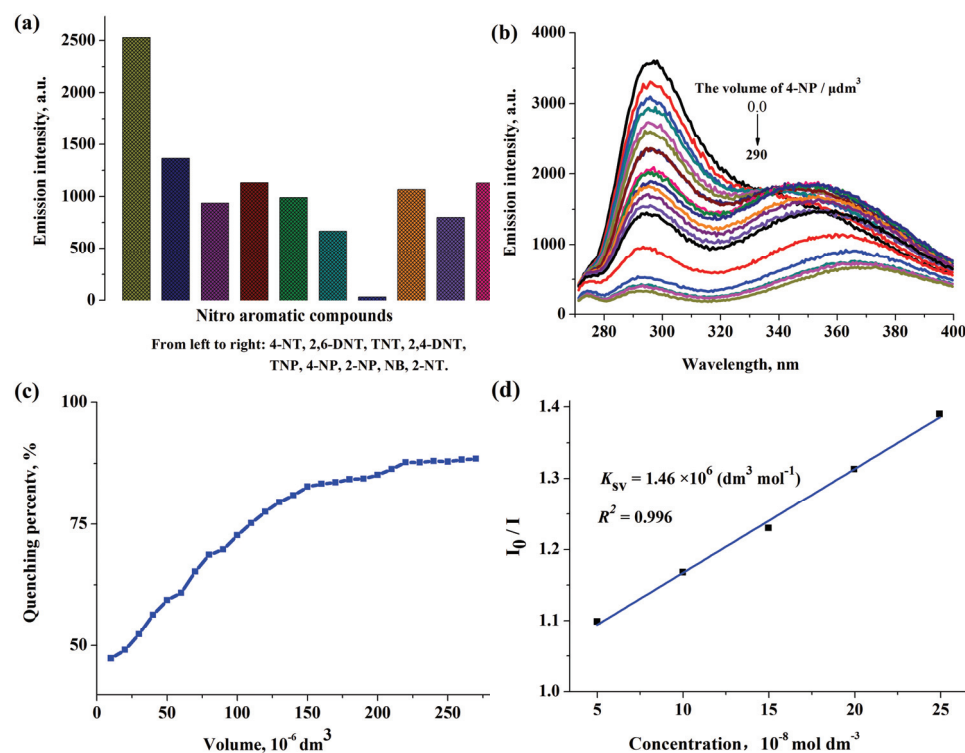

Fig. 3. a) Fluorescence spectra bar graph representation of the probe solution upon addition of various NACs, b) fluorescence spectra changes of the probe solution upon addition of different amounts of 4-NP, c) quenching percentages representation of the probe solution upon addition of different volumes of 4-NP and d) Stern–Volmer plot for the quenching of 4-NP.

TABLE I. Comparison of analytical performance for p-NP using the fluorescence methods

Probe	LOD, µmol dm^{-3}	K_{sv} / dm^3 mol^{-1}	Ref.
Histidine	3.5	6.1×10^4	27
Polymer carbon dot	0.26	1.8×10^4	28
[Tb(BTEC)$_{0.5}$(HCOO)(H$_2$O)$_2$]	0.0003	2.0×10^4	29
PEI stabilized AgNCs	1.28	8.7×10^4	30
Cu-doped carbon dots	5.8	6.4×10^3	31
{[LnxLn'1–x(HL)(H$_2$O)$_2$]·(H$_2$O)$_2$}$_n$	0.98	1.9×10^5	32
Polymeric probe	0.028	1.5×10^6	This work

Fluorescence detection of Fe^{3+}

Next, the fluorescence quenching behavior of poly(NIPAM–AM–BMPS) in the presence of various metal ions (1.0 mol m^{-3}, λ_{ex} = 295 nm), such as Na$^+$, Mg^{2+}, Li$^+$, Ca^{2+}, Ag$^+$, Cr^{3+}, Zn^{2+}, Cd^{2+}, Ni^{2+}, Cu^{2+}, Co^{2+}, Mn^{2+}, Ba^{2+}, Al^{3+} and Fe^{3+}, was studied. Only Fe^{3+} caused an obvious fluorescence quenching as shown in Fig. 4a. Therefore, poly(NIPAM–AM–BMPS) has a good fluorescence response towards Fe^{3+} but not towards the other metal ions. A gradient titration experiment was performed by adding different volumes of Fe^{3+} solution (1.0 mol m^{-3}) to an aqueous solution of poly(NIPAM–AM–BMPS). With the increasing volume of Fe^{3+} solution, the fluorescence emission intensity at 293 nm decreased gradually without an obvious shift in the wavelength as seen in Fig. 4b. According to the fluorescence intensity, the quenching efficiency was evaluated. The Stern–Volmer constant K_{SV} of poly(NIPAM–AM–BMPS) toward Fe^{3+} was determined by Stern–Volmer equation based on the fluorescence titration results. A good linear dependence of the fluorescence intensity I_0/I on the Fe^{3+} con-
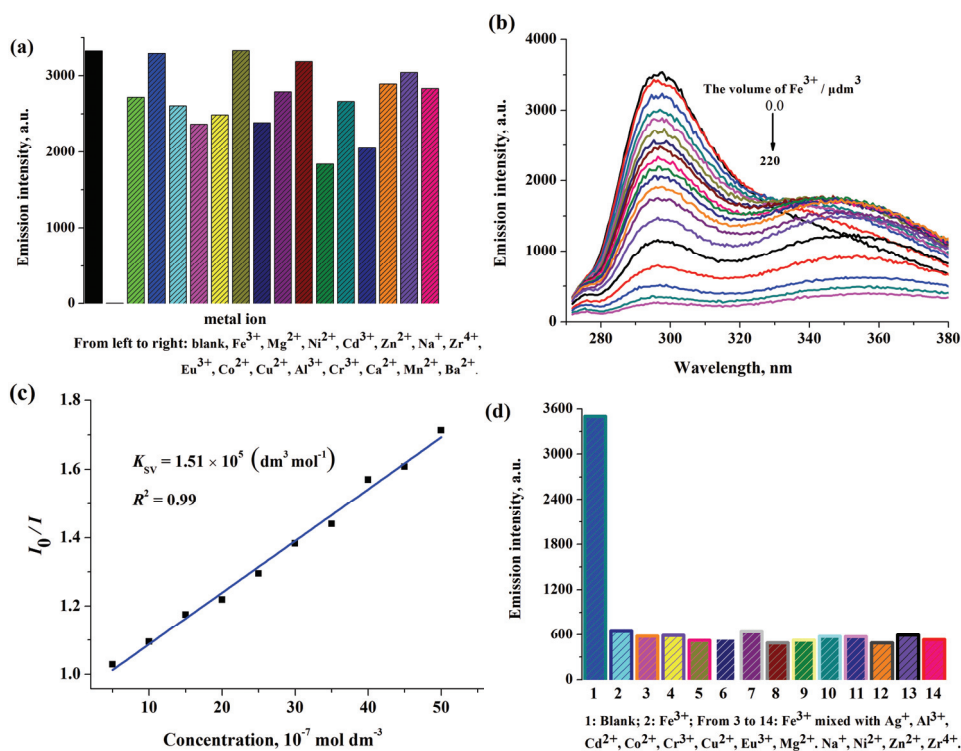

Fig. 4. a) Fluorescence spectra bar graph representation of the probe solution upon addition of various metal cations, b) fluorescence spectra changes of the probe solution upon addition of different amounts of Fe^{3+}, c) Stern–Volmer plot for quenching of Fe^{3+} and d) fluorescence intensities of the probe solution toward various metal ions.

centration in the range of $(5.0-50.0)\times10^{-7}$ mol dm^{-3}was obtained ($R^2 = 0.99$), Fig. 4c. The corresponding constant K_{sv} for Fe^{3+} probing was calculated to be 1.51×10^5 dm^3 mol^{-1}, and the corresponding *LOD* (limit of detection) value was 2.2×10^{-7} mol dm^{-3}. The results showed that poly(NIPAM–AM–BMPS) has high sensitivity for fluorescent sensing of Fe^{3+} in aqueous solution. Possible reasons for the fluorescence quenching of the polymer when adding various metal ions was summarized in a previous reports.[33] It is most likely caused by the energy or electron-transfer reactions between the polymer backbone and the binding metal ion, which is a nonradiative center and trapped the excitation energy passing through them.[34] To gain further insight into the influence of other metal ions on the interaction between poly(NIPAM–AM–BMPS) and Fe^{3+}, competitive experiments in the presence of the other tested metal ions were performed.

Fluorescence quenching occurred when Fe^{3+} was added to a blank aqueous solution of poly(NIPAM–AM–BMPS), Fig. 4d. Additionally, when 1.0 equiv. of various metal ions was added to the polymer solution containing Fe^{3+}, fluorescence quenching was also observed. Therefore, the presence of co-existing metal cations had almost no remarkable influence on the fluorescence intensity of probe toward Fe^{3+}. These results showed that poly(NIPAM–AM–BMPS) can sensitively and selectively detect Fe^{3+} in aqueous solution. Furthermore, the performances of poly(NIPAM–AM–BMPS) were compared with those of other fluorescent probes (Table II).[35–40] The comparisons showed that the present fluorometric strategy could exhibit better sensitivity performances for Fe^{3+} detection.

TABLE II. Comparison of analytical performance for Fe^{3+} sensing using fluorescent methods

Probe	*LOD* / μmol dm^{-3}	Linear range, μmol dm^{-3}	Ref.
Polydopamine	3.7	12.5–62.5	35
9-Dimethoxymethyl-anthracene	3.08	0–90	36
PVA-pyrene	0.73	0–90	37
Au nanoclusters	3.50	5.0–300	38
Benzimidazolium probes	2.80	5.0–1280	39
Graphene Q-dots	7.22	350–2700	40
Polymeric probe	0.22	0.5–5.0	This work

CONCLUSIONS

In summary, a novel water-soluble polymer containing diphenyl sulfone-functionalized units has been successfully designed and synthesized. It allows for a fast-response and sensitive fluorescence detecting of Fe^{3+} with a low detection limit of 0.22 μmol dm^{-3}. In addition, this polymer also shows an excellent sensing performance towards TNP with good selectivity and a low detection limit of 0.028 μmol dm^{-3}, suggesting its good future practical applications.

И З В О Д

СИНТЕЗА, КАРАКТЕРИЗАЦИЈА И СТУДИЈА ФЛУОРЕСЦЕНТНИХ ОСОБИНА НОВОГ ДИФЕНИЛ СУЛФОН-ФУНКЦИОНАЛИЗОВАНОГ ПОЛИМЕРА РАСТВОРЉИВОГ У ВОДИ

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У раду је приказана синтеза новог дифенил сулфон-функционализованог полимера растворљивог у води лаким хидротермалном синтезом, који је затим је окарактерисан FT-IR, UV–Vis, ^{1}H-NMR and ^{13}C-NMR спектрометријама. Експерименти са гашењем (квенчовањем) флуоресценције открили су да су интензитети флуоресценције добијеног дифенил сулфон-функционализовано полимера линеарни зависни од концентрација Fe^{3+} и 4-нитрофенола (4-NP) у опсегу концентрација $(5,0-24,9)\times10^{-8}$, односно $(5,0-50,0)\times10^{-7}$ mol dm^{-3}, са границом детекције $2,8\times10^{-8}$ односно $2,2\times10^{-7}$ mol dm^{-3}. Ови резултати отварају могућност за развој нових хемосензора увођењем селективне флуоресцентне групе у полимерне материјале.

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