



Article Chiral Binaphthol Fluorescent Materials Based on a Novel Click Reaction

Fuchong Li¹, Wei Liu², Li Tian¹, Wei Cao², Xu Li³, Junhong Guo¹, Jinfeng Cui¹ and Baoping Yang^{1,*}

¹ School of Petrochemical Engineering, Lanzhou University of Technology, Lanzhou 730050, China

- ² Key Laboratory of Special Function Materials and Structure Design, Institute of National Nuclear Industry, Frontiers Science Center for Rare Isotope, School of Nuclear Science and Technology, Ministry of Education, Lanzhou University, Lanzhou 730000, China
- ³ The College of Chemistry and Chemical Engineering, The Northwest Normal University, Lanzhou 730070, China
- * Correspondence: yangbaoping2004@163.com

Abstract: Because of easy functionalization, low cost, and large-scale fabrication, pure organic fluorescent polymers are widely applied in light-emitting display, bio-fluorescence-enhanced imaging, explosive detection, and other fields. Among these applications, due to their unique optical rotation characteristics, chiral fluorescent polymer materials are part of fluorescent polymers which could be used in chiral molecular detection and separation, biological target detection, etc. In this work, we designed and synthesized the first chiral organic fluorescent polysulfate materials through sulfur fluoride exchange polymerization (new click chemistry) by asymmetric binaphthol molecular. The chiral fluorescent polysulfate were synthesized by R/S [1,1'-binaphthalene]-2,2'-diol(Binol.), propane-2,2-diylbis(4,1-phenylene) bis(sulfurofluoridate) (FO₂S–BA–SO₂F) and 4,4'-(propane-2,2-diyl)diphenol(BA.) through step-by-step polymerization reaction under alkali present. It was found that the local crystallization of pure bisphenol A polysulfate was broken by the asymmetric axial chiral BINOL molecule inserted in it and let the polymer into the amorphous state. Fluorescent chiral molecules are uniformly dispersed in the polymer; the 120 μ m film prepared by the film scraper was transparent and had good luminescence characteristics under ultraviolet light. After fluorescence detection, the excitation wavelength is 450 nm, and the emission wavelength is 480 and 517 nm.

Keywords: polysulfate material; fluorescence; polymer; chiral binaphthol

1. Introduction

Chiral fluorescent sensors are the molecules or materials which can interact with the other chiral targets and convert the enantioselective recognition events into observable optical signal outputs, such as fluorescence quenching/enhancement, ratiometric changes, fluorescence lifetime changes, etc. Especially design and synthesis of optical sensors relates to the chiral host–guest chemistry in combination with molecular recognition have been very important in recent years. Binaphthol (BINOL) and its derivatives, which have an asymmetry C2 axis, are a class of chiral molecules applied to prepare molecular-fluorescence agents [1–3], such as Fe (III) recognition, and host–guest interactions research. These new materials combined with functional small molecules and polymers can be designed and synthesized using local chiral structures to realize detection and chiral separation [4–9].

Chiral fluorescent polymer materials have become more and more interesting to scientists because of their excellent device processability and recoverability compared with small molecules. The scientists focus on how to introduce the chiral fluorescent molecules into polymer chains to form different topological and micro-chain structures. Until now, there are two typical methods to construct chiral polymeric fluorescent materials. One is called "post-modification" which uses chemical and physical methods to graft fluorescent groups onto the end or side chain of the polymer. The second method, called "chemical



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). copolymerization", uses chiral fluorescent as the third functional monomer to make chiral fluorescent polymers by mixing with the main monomers. In addition to the design and synthesis of chiral fluorescent groups with specific luminescent properties, the development of new polymers with special chemical and physical stability—especially those with good strength in complex chemical environments—is also an urgent problem to be solved.

Since the click reaction was first reported by Karl Barry Sharpless's group in 2001 year [10], more and more scientists have been trying to figure out how to make and use other click chemical reactions to make small molecules and polymers. The sulfur-fluoride exchange reaction is one efficient click reaction discovered in recent years [11,12]. Due to the sulfate structure formed, the sulfur fluoride exchange reaction has the characteristics of high efficiency, mild reaction conditions, and stable chemical properties. In recent years, scientists and engineers have invested great interest in sulfate materials. Due to its good pollution resistance, sulfate materials have successfully prepared membranes for wastewater treatment [13–15], which could be inferred to replace commercial materials such as polysulfone and PVDF. SuFEx click chemistry was also used to synthesize polysulfate-bound MacMillan catalysts and selectivity in the asymmetric Diels-Alder (DA) reaction [16]. Furthermore, there are few reports on how to design and make polysulfate materials with fluorescent properties through sulfur-fluoride exchange copolymerization directly, especially sulfate fluorescent materials with a chiral structure, which is needed to make membranes and catalysts for wastewater treatment. Only one paper reported the fluorescent materials prepared by the post-modification method by clicking sulfur–fluorine exchange reaction [17]. Therefore, it is essential to expand the application of sulfate materials and study the luminescent properties of fluorescent copolymer materials. Chiral binaphthol structure and its derivatives often design and synthesize materials with fluorescence properties and chiral structure, such as Zn (II)-coordination fluorescence enhancement binaphthol polymer [18–21]. However, as far as I know, no paper has reported how to construct sulfate materials with stable chemical properties by binaphthol molecule, especially designing chiral fluorescent polymers using binaphthol structure as the main monomer for copolymerization through sulfur-fluoride exchange reaction. We introduced R/S binaphthol into the polysulfate material skeleton through a sulfur–fluoride exchange copolymerization reaction so far in this work. We studied polymer and property influence (Figure 1) resulting from the different amounts of binaphthol were added to the fluorescent polysulfate. It lays the foundation for future research for preparing luminescent materials from polysulfate polymers, especially fluorescent chiral polymer devices with bio-targeted detection and separation.



(a)

Figure 1. Cont.



Figure 1. (a) Photos of chiral binaphthol polysulfate fluorescent polymers under ultraviolet light; (b) Synthesis of chiral binaphthol polysulfate fluorescent polymers.

2. Materials and Methods

Bisphenol A (BA) and sodium carbonate (Na₂CO₃) were purchased from Aladdin Chemical Co., Ltd. (Beijing, China), Propane-2,2-diylbis (4,1-phenylene) bis (sulfurofluoridateFerric) [BA–(SO₂F)₂] was purchased from Baiyin TW company, china, R–BINOL, S–BINOL, DMF, MeOH, THF, and chloroform were supplied by Sinopharm Chemical Reagent Co., Ltd., Shanghai, China, polyvinyl alcohol (pva 1788) was purchased from Wuhan Huaxiang Kejie Biotechnology Co., Ltd., Wuhan, China. All organic solvents used are analytical grade and without further purification. Lanzhou Chemical Research Center, China, prepared deionized water.

2.1. Preparation of Polymer: PSE-R/S-BINOL50%

The chiral binaphthol polysulfate fluorescent polymers PSE-R-BINOL50% was synthesized as Figure 2. The method is following: the solution of R–BINOL (0.05 mol, 14.6 g), $BA-(SO_2F)_2$ (0.051 mol, 20.0 g), and Na_2CO_3 (0.11 mol, 11.6 g) in 50 mL DMF was replaced with nitrogen three times and stirred at room temperature for 15 min. Then the solution temperature was gradually increased to 140 °C in 30 min. The solution was stirred at 140–145 $^{\circ}$ C for another 6–8 h. After the viscosity of the reaction mixture reached a certain degree, the mixture was cooled to 100 °C and poured into hot deionized water (60 $^{\circ}$ C) for precipitation to get a white solid crude product. Then the crude product was dissolved in chloroform and precipitated with ethanol three times. Finally, the pure white powder was obtained by being extracted with hot deionized water and ethanol for 24 h, and the pure product was dried in a vacuum for 5 h to obtain the solid white powder PSE-R-BINOL50% (28.0 g, yield 86%), PSE molecular weight: Mn = 172,771 g/mol (Figure S2). Figure S1 The Nuclear magnetic resonance spectrum of the PSE–R–BINOL50%. Figure S2 shows the molecular weight by polymer gel chromatography. XPS diffraction in Figure S3 showed that oxygen, carbon, and sulfur peaks are, respectively, which proved that the polymer forms the sulfate structure. The polymer is dissolved in chloroform and tested by the circular dichroism spectrum (CD spectrum). Figure S4 shows that the material shows certain optical rotation characteristics, which proves that introducing chiral molecules can build a chiral polysulfate polymer. ¹H-NMR (CDCl₃, 400 MHz) δ H: 8.03~8.05(m,2H), 7.92~7.94(m,2H), 7.84~7.86(m,1H), 7.72~7.77(m,1H), 7.59~7.66 (m,3H), 7.43~7.51 (m,4H), 7.27~7.41(m,3H), 6.93~7.21(m,9H), 6.74~6.89(m,2H), 6.54~6.68(m,2H), and 1.46~1.56(m,9H). ¹³C NMR (100 MHz, CDCl3) δ:149.56, 149.51~149.49, 146.80~146.46, 133.2~131.02, 128.78~126.46, 121.26~120.65, 120.62,120.06, 120.03, 119.13, 42.75~42.59, and 30.79~30.74.



Figure 2. Synthesis of chiral binaphthol polysulfate fluorescent polymers PSE–R–BINOL50%.

The polymer PSE–S–BINOL50% was synthesized similarly to the PSE–R–BINOL50% above. The yield of the polymer was (26.7 g, yield of 82%), PSE molecular weight: Mn = 43,648 g/mol (Figure S2).

2.2. Preparation of PSE-R-BINOL1%

The chiral binaphthol polysulfate fluorescent polymers PSE–R–BINOL1% was synthesized following a literature procedure as Figure 3. The solution of R–BINOL (1 mmol, 0.286 g) and BA (99 mmol, 22.572 g) in 90 mL DMF was stirred with a mechanical agitator at 170 °C in the oil bath. Then the BA–(SO₂F)₂ (100 mmol, 39.2 g) was added to the mixture and Na₂CO₃ (22 mmol, 23.32 g) was added. The mixture appeared with lots of CO₂ bubbles and was stirred at this temperature for another 6–8 h. After the viscosity of the reaction mixture reached a certain degree, the mixture was cooled to 100 °C and poured into hot deionized water (60 °C) for precipitation to get a white solid crude product. Then the crude product was dissolved in chloroform and precipitated with ethanol 3 times. Finally, the white powder was obtained by being extracted with hot deionized water and ethanol for 24 h. The pure product was dried in a vacuum for 5 h to obtain the solid white powder PSE–R–BINOL1% (52.8 g, yield 91%), PSE molecular weight: Mn = 33,937 g/mol. ¹H-NMR (CDCl₃, 400 MHz) δ H:7.32~6.70(m, 1H), 1.82~1.50(m, 1H). ¹³C NMR (400 MHz, CDCl₃) δ : 150.03~148.36, 128.40, 120.65, 42.78~42.76, 30.80.



Figure 3. Synthesis of chiral binaphthol polysulfate fluorescent polymers PSE-R-BINOL1%.

2.3. Device Fabrication Process of PSE–R/S–BINOL50%

2.3.1. Preparation of Fluorescent Transparent Film: PSE-R/S-BINOL50%

The fluorescent transparent film fabrication process of PSE–R/S–BINOL50% is shown in Figure 4. The white PSE–R/S–Binol polymer powder (2 g) was dissolved in THF 10 mL. The solution was stirred vigorously to form a uniform polymer gel solution. The gel solution was put on the desk for a few minutes until the bubbles basically disappeared, and then put the mixture of 2 mL was on the PET release film, then used a film scraper to scrape the film, put the coated film for another 5 min, put the coated film in a vacuum oven to dry another 10 min at 70 °C, the PSE–R/S–Binol polymer film was separated from the PET release film to obtain PSE fluorescent transparent film.







PSE-R/SBINOL50%

PSE-R/SBINOL50% Film

Figure 4. Fabrication process of PSE–R/S–BINOL50%.

2.3.2. Preparation of Microspheres: PSE-R/S-BINOL50%

PSE–R/S–Binol polymer powder (1 g) was dissolved in dichloromethane 8 mL as the oil phase. The oil phase was then added into 50 mL pva (Mn = 8000 g/mol, 1% aqueous solution), the mixture was ultrasonic for another 10 min, stirred at room temperature for another 12 h, and the product obtained by centrifuge. The microspheres were washed with ethanol, and deionized water and then dried in a vacuum oven to obtain solid microsphere powder. The powder was dispersed in ethanol and dropped on the silicon chip to observe its SEM. The SEM showed that in Figure 5 below. Both R/S polymers could be emulsified to form microsphere particles. 100 microsphere was selected for size measurement in Figure 5. We found that the size of the microspheres of more than 80% was between 0.78 μ m and 1.56 μ m prepared by this method. The microsphere distribution of S–BINOL is slightly wider than that of R–BINOL, which the molecular weight difference may cause. This method lays a good foundation for the polymer to prepare fluorescent microspheres and provides a good path for biological fluorescence detection in the future.



Figure 5. SEM of the PSE-R/S-BINOL50% polymer microspheres and size distribution.

3. Results and Discussion

3.1. Structure Descripition

A new kind of chiral polysulfate material was synthesized by a new click reaction using bisphenol A skeleton and binaphthol skeleton. The chemical structure information of the material was determined by nuclear magnetic resonance, IR, gel permeation chromatography, and circular dichroism spectrum.

According to the molecular structure simulation of the polysulfate polymer through gauss view software in Figure 6 showed that the structure of sulfate bond $-O-S(=O)_2-O-$, which is constructed by sulfur (VI) is similar tetrahedron. The stable sulfur (VI) structure

endows the polymer with strong chemical stability, including resisting the extreme chemical environment, such as acid and alkali.



Figure 6. Schematic diagram of molecular structure of PSE-BINOL and BisphenolA PSE.

In this work, asymmetry C2 axis molecular BINOL was copolymerized into BisphenolA PSE to break its regularity. As shown in Figure 6, the structure of bisphenol A copolymerized with R/S–BINOL (mole ratio = 1:1) becomes a twisted one-dimensional chain simulated with gaussian software. Even when a small amount of BINOL monomer (BA:BINOL = 99:1 (mole ratio)) is inserted into the BisphenolA PSE polymer chain, it could not change the order state of local polymer segments. However, it could also change the polymer's long-range order and 3D stacking state.

The structure and fluorescence properties of the PSE materials were described in detail in partial characterization as follows. Figure 7 (infrared spectrum) showed that wavenumber 2969 cm⁻¹ and 2890 cm⁻¹ were the stretching vibrations of the isopropyl methyl CH₃ in the bisphenol A structure. The C=O vibration peak of a small quinone structure in bisphenol A was 1880 cm⁻¹, and 3335 cm⁻¹ was the hydroxy OH peak of bisphenol A in the broad package.



Figure 7. Infrared spectrum of the PSE-R-BINOL50% and reagents.

The blue line shows the infrared spectra of the monomers shown in Figure 7 (bisphenol A, binaphthol, $FO_2S-BA-SO_2F$) and PSE-R-BINOL50% polymer represented the infrared spectrum of $FO_2S-BA-SO_2F$ monomer, which was obtained by the reaction of bisphenol A and sulfuryl fluoride under organic base present. Therefore, the signal near 3335 cm⁻¹ disappeared in the infrared spectrum while the signal at 1904 cm⁻¹ may be the signal of

the appeared acyl halide structure. It is speculated that this is the vibration of $(O=)_2S$ -F structure, and it is proved that the structure of FO₂S–BA–SO₂F. In addition, compared with the binaphthol, the signal of PSE–R–BINOL50% materials near 3432 and 3054 cm⁻¹ proved that the binaphthol structure had been successfully introduced into BisphenolA PSE. Compared with the infrared spectrum of PSE–R–BINOL50%, the signal near 3065 cm⁻¹ of PSE–R–BINOL1% in Figure 8 showed that there was less amount of binaphthol molecules copolymerized into the BisphenolA PSE.



Figure 8. Infrared spectrum of the PSE-R-BINOL1% and PSE-R-BINOL50%.

We know that the bisphenol A PSE (Figure 6) is a kind of polymer with part crystallinity. In this work, we introduced asymmetry C2 axis molecular binaphthol into the bisphenol A PSE to destroy its molecular regularity, which could change its thermodynamic properties. From the thermodynamic curve in Figures 9 and 10 below: while just a few binaphthol monomers (1% molar of bisphenol A) were copolymerized into the bisphenol A PSE, the decomposition temperature of the PSE-R-BINOL1% was about 330 °C and its glass transition temperature was about 171 °C that was higher than the glass temperature of bisphenol A PSE. When the copolymerization ratio of the binaphthol was increased to 50% (bisphenol A:binaphthol = 1:1 (mole ratio)), the glass transition temperature and the decomposition temperature decreased significantly (Figures 9 and 10). The decomposition temperature curves of PSE–R–BINOL50% and PSE–S–BINOL50% are basically the same after the R/S binaphthol copolymerization into bisphenol A PSE. Simulation was based on thermodynamic data and polymer molecular chain model in Figure 6. It might be inferred that when less binaphthol copolymerized into the bisphenol A PSE, it could slightly damage the crystalline state of bisphenol A PSE and the large chiral steric structure would entangle the bisphenol A PSE molecular chains. Therefore, the glass transition temperature was increased. When a large amount of binaphthol copolymerized into the bisphenol A PSE molecular chain, the regular structure was almost completely broken, and the polymer could become an amorphous state. Thereby, the glass transition temperature was significantly reduced. In addition, when the number of binaphthol molecules copolymerized into the bisphenol A PSE molecular chain increased, disordered molecular stacking could produce more hydrolysis holes which might affect the polymers' chemical and thermal stability. Figure 9 displayed that the thermal decomposition temperature of PSE–R/S–BINOL50% was lower than that of PSE–R–BINOL1%.

Due to the building blocks (bisphenol A and BA–(SO₂F)₂) and configuration of the sulfate ester linker, the bisphenol A PSE existed in a certain long-range order. In fact, the stacking between the bisphenol A PSE molecular chains has a certain regularity shown by PXRD (Figure 11). In order to prove that there was no residual reactive monomer in the polymer, the X-ray powder diffraction of the polymers (PSE–R/S–BINOL) compared with the monomers (bisphenol A, binaphthol, FO₂S–BA–SO₂F) in the Figures 11 and 12. It could be clearly seen that there was no monomer existed in the polymer. Compared

with the bisphenol A PSE, PSE–R/S–BINOL became an amorphous state polymer. Chiral binaphthol molecules would destroy the crystallization of the bisphenol A PSE material and change its thermodynamic properties.



Figure 9. Thermal decomposition curve of PSE.



Figure 10. Heat flow curve of PSE.



Figure 11. X-ray powder diffraction of the PSE polymer.

3.2. Photoluminescence Properties

By the chiral molecular R/S-binaphthol monomer introduced into bisphenol A PSE polymer, the order of molecular chains could be destroyed and the amorphous state of the polymers can be increased.

In this work, transparent fluorescent film was prepared by solvent method as shown in Figure 13. The luminescence properties of PSE–R–BINOL50% in the solid state at room temperature were evaluated. Under 254 nm UV light, the photos show that the PSE– R–BINOL50% emit strong blue fluorescence. After the large steric hindrance monomer (binaphthol) enters the bisphenol A PSE polymer, the polymer became transparent under natural light, this work provided a new method for the new transparent fluorescent materials preparation in the future. As shown in Figure 14, PSE–R–BINOL50% was exhibited at 450 nm, the luminescence spectrum of PSE–R–BINOL50% exhibited two distinct characteristic emission peaks at 480 and 517 nm.



Figure 12. X-ray powder diffraction of PSE polymer and monomers.



PSE-RBINOL50%

Figure 13. Luminescence of the PSE-R-BINOL50% under 254 nm.



Figure 14. (a) Excitation spectrum and (b) luminescence spectrum of the PSE–R–BINOL50%.

4. Conclusions and Outlook

Through a new type of click reaction (sulfur–fluorine exchange polymerization), we successfully introduced the C2 axis chiral molecule BINOL into the bisphenol A PSE polymer to synthesize chiral fluorescent polysulfate. Less (1%) of binaphthol molecular copolymerization into bisphenol A PSE could increase the glass transition temperature and break the molecular order of the bisphenol A PSE. When the polymer has transformed into an amorphous polymer, the blue fluorescent transparent film was successfully prepared by solvent evaporation. We have successfully designed a film that was excited and emitted by visible light (excitation wavelength was 450 nm, emission wavelength was 480 nm and

517 nm). This work is the first chiral fluorescent polymer designed and synthesized in the field of new polysulfate materials and the first research work on exploring micro-scale microspheres, which will significantly expand the application of polysulfate materials for fluorescence detection and chiral separation devices preparation in the future.

Supplementary Materials: The following supporting information can be downloaded at: https: //www.mdpi.com/article/10.3390/sym15030629/s1. Figure S1: The Nuclear magnetic resonance spectrum of the PSE–R–BINOL50%; Figure S2: Molecular weight by polymer gel chromatography; Figure S3: The XPS diffraction of PSE–R–BINOL50%; Figure S4: CD spectrum of PSE–R–BINOL50%.

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