

# Synthesis and Characterization of Poly(1-methoxy-4-octyloxy)-Para-Phenylene Vinylen for Light-Emitting Diodes Application

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# ABSTRACT

In this study, the conjugated polymer, poly(1-methoxy-4-octyloxy)-*para*-phenylene vinylene (MO-*p*-PPV) was synthesized and characterized. MO-*p*-PPV was synthesized according to Gilch polymerization mechanism by using 4-methoxyphenol as starting material in the presence of potassium tert-butoxide (1M in THF). The product was further purified by multiple precipitations in different solvents such as methanol, tetrahydrofuran, isopropyl alcohol and hexane. The final product was dried to afford MO-*p*-PPV as a red solid. The resulting polymer was completely soluble in common organic solvents. The structure of monomer and optical properties of polymer were characterized by proton nuclear magnetic resonance (<sup>1</sup>H-NMR) spectroscopy, UV-vis spectroscopy, and fluorescence spectroscopy. The UV-vis spectrum showed absorption maxima for MO-*p*-PPV at 491 nm. Similarly, fluorescence spectrum showed  $\lambda_{max}$  emission at 540 nm.

Keywords: Component; Poly(1-Methoxy-4-Octyloxy)-Para-Phenylene Vinylen; Gilch Polymerization; Light-Emitting Diodes

## 1. Introduction

During the past decade, an explosive growth of activity in the area of organic electroluminescence has occurred in both academia and industry [1]. As the potential base material in organic light-emitting diodes (OLEDs), conjugated polymers have been widely explored. For example, since the discovery of electroluminescence in poly(p-phenylene vinylene) (PPV) [2,3], a wide variety of conjugated and semi-conjugated polymers have been used as the active emissive layer in OLED devices [4-7]. Polymer light-emitting diodes (PLEDs) are promising devices, especially for next generation active matrix displays. Solution deposition techniques, homogeneous large area thin films, reduced manufacturing process complexity, low-cost, high luminescence efficiency, large spectral range, and relatively simple device structures are some of the main reasons for an increased interest in polymer materials for LED's []. M. T. Bernius, M. Inbasekaran, J. O'Brien, and W. Wu, Adv. Mater., 12, 1737 (2000).

We report here the preparation of poly(1-methoxy-4-octyloxy)-para-phenylene vinylene (MO-p-PPV) for light emitting diodes application by using Gilch polymerization route. A typical procedure for the synthesis was described in the Experimental section.

## 2. Experiment

#### 2.1. Materials

4-methoxyphenol, 1-bromooctane, Sodium bromide, paraformaldehyde, potassium tert-butoxide (1 M solution in THF), KOH, glacial acetic acid, conc.  $H_2SO_4$  were purchased from Aldrich Chemical Co. and used without further purification unless otherwise noted. THF was dried and purified by fractional distillation over sodium/benzophenone and handled in a moisture-free atmosphere.

#### 2.2. Measurements

<sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded using a Bruker avance 400 MHz, and chemical shifts were recorded in ppm. The data were processed using NUTS NMR Utility Transform Software (Acron NMR). The UV-vis spectra were recorded on a Perkin Elmer Lambda 19 UV-VIS-NIR spectrophotometer with baseline corrections and normalizations carried out using WinLab software. Fluorescence spectra were collected on a Perkin Elmer Luminescence Spectrometer LB 50.

4-methoxyphenol (10.0 g, 0.083 mol) was dissolved by 100 ml ethanol, 6.0 g (0.12 mol) of KOH and octyl bromide (22.4 g, 0.12 mol) were added and strired at 70  $^{\circ}$ C for 24 h. After the reaction, precipitate was collected by filtration and washed with ethanol. White crystalline 1 (67% yield) was obtained.

*1,4-Bis(bromomethyl)-methoxy-4-octyloxy benzene* (2)

Methoxy-4-octyloxy benzene (0.169 mol), Sodium bromide (0.097 mol), paraformaldehyde (0.166 mol) were dissolved in 24 ml of glacial acetic acid. 50% conc.  $H_2SO_4$  in glacial acetic acid was added and the reaction was heated at 70°C for 24 h. Then saturated NaHCO<sub>3</sub> was added until the red color disappeared. The mixture was extracted three times with dichloromethane. The organic extracts were combined, washed with brine, and dried with magnesium sulfate. Upon filtering the solution and evaporating the solvent, a white solid was obtained, which was recrystallized in hexane and washed with cool me-

thanol to give white crystals of pure 2 (60%).

Poly(1-methoxy-4-octyloxy)-para-phenylene vinylene (MO-p-PPV)

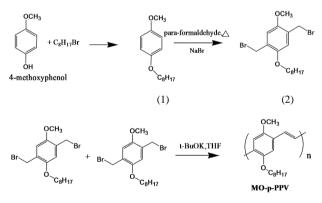
of 1,4-Bis(bromomethyl)-methoxy-4-octyloxy benzene (0.00238 mol) was dissolved in 25 mL of anhydrous THF under nitrogen, and a 10 mL of THF solution of potassium tert-butoxide was added. The mixture was stirred overnight at room temperature under nitrogen. The resulting polymer was precipitated into 100 mL of methanol, and the mixture was centrifuged. The supernatant was decanted, and the residue was re-dissolved in a minimum amount of THF. The crude polymer was then successively re-precipitated into methanol and propanol. Red crystal-line (55%) was obtained

## 3. Results and Discussion

**Figures 1** and **2** shows the UV-Vis absorption and fluorescence (FL) spectra of the MO-*p*-PPV synthesized via Gilch polymerization. The Gilch polymerization affords MO-*p*-PPV having a broad absorption band with  $\lambda_{max} = 491$  nm. This band can be attributed to  $\pi$ - $\pi$ \* transitions of the conjugated backbones. Fluorescence spectra obtained upon excitation at 497 nm. The Gilch polymerization affords MO-*p*-PPV having a strong emission band at 540 nm.

#### 4. Conclusions

Poly (1-methoxy-4-octyloxy)-*para*-phenylene vinylene (MO-p-PPV) was successfully synthesized by Gilch polymerization. UV-Vis absorption spectrum in THF showed the broad absorption band with  $\lambda_{max} = 491$  nm. The PFV exhibited a greenish fluorescence at  $\lambda_{max}$  emission = 540 nm.



Scheme 1. Synthesis of poly(1-methoxy-4-octyloxy)-para-phenylene vinylene (MO-p-PPV).

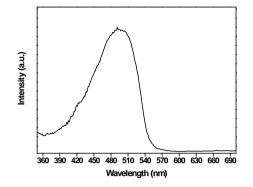


Figure 1. UV-Vis spectrum of poly(1-methoxy-4-octyloxy)-*para*phenylene vinylene (MO-*p*-PPV) (1.0 x  $10^{-4}$  M) prepared by Gilch polymerization in THF at room temperature.

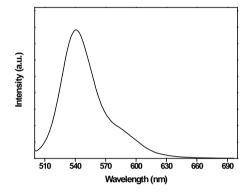


Figure 2. Fluorescence spectrum of Poly (1-methoxy-4-octyloxy)para-phenylene vinylene (MO-p-PPV) (1.0 x 10<sup>-5</sup> M) Gilch polymerization in THF at room temperature (excitation 497 nm).

#### 5. Acknowledgements

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