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Synthesis and Characterization of C_{60}-Containing Poly(ethylene oxide)

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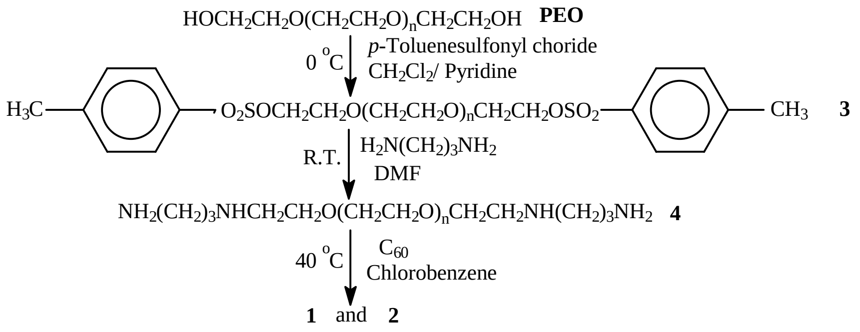
Abstract: C_{60}-Containing poly(ethylene oxide) (PEO) was synthesized by a new method. Molecular structural characterization for the polymers was presented by ^{1}H-NMR, infrared and UV-Vis spectra.

Keywords: PEO, C_{60}, synthesis.

C_{60} has attracted much attention due to its unique properties[1, 2], and the grafting of biocompatible polymers onto C_{60} is of special interest due to the potential importance of the fullerene molecule in the biomedical and biotechnological fields[3]. Poly(ethylene oxide) (PEO) is well known for its remarkable biomedical properties, and some C_{60}-containing PEO have been prepared[4, 5]. However, the reported methods were not so easy and the reaction conditions were not mild.

Recently we have developed a new method to synthesize C_{60}-end-capped PEO; the method was very easy and the reaction conditions were mild. Herein, we would like to report the synthetic procedure and structural characterization of the end product.

Scheme 1



The synthetic route was shown in **Scheme 1**. First, PEO ($M_w = 20000$)was reacted with *p*-toluenesulfonyl chloride to yield **3**, which was then converted to a precursor PEO

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possessing amino end-groups **4** in DMF in the presence of 1, 3-diaminopropane. At last **4** reacted with C_{60} in chlorobenzene to give the C_{60}-containing PEO (**1** and **2**), which were purified by several precipitations from chloroform into methanol.

Results and Discussion

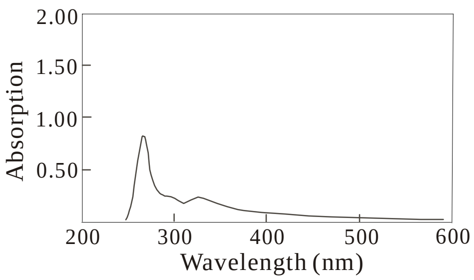
1 and **2** have good solubility in common organic solvents, such as $CHCl_3$, THF, DMSO and DMF, *etc*., and also they are soluble in water. The UV-Vis spectrum of **1** in chloroform was shown as an example in **Figure 1**. The two peaks at 269 nm and 330 nm were attributed to the absorption of C_{60}. This spectrum confirmed that C_{60} was successfully covalently linked to PEO, since C_{60} was not soluble in chloroform[6]. The C_{60} concentrations (w/w) in **1** and **2** were 0.9 % and 1.5 %, respectively, which were determined by comparing the intensity of absorption peak at 306 nm in toluene with that of pure C_{60} in toluene.

In the IR spectrum of **1** and **2**, two new absorption peaks appeared apparently at 527 and 576 cm^{-1}. While **4** did not absorb in this region, these results further proved that C_{60} had covalently bonded to the PEO backbone[7].

The signals of the phenyl ring of **3** in the ^1H-NMR spectra had completely disappeared in **4**, **1** and **2**. This confirmed that the *p*-toluenesulfonyl groups were replaced by the amino moieties completely. And some new peaks appeared at δ2.94, 2.87 and 1.62 ppm in ^1H-NMR of **4,** which were assignable to the protons of the propanediamino groups.The new weak peak at 3.48 ppm attributed to the resonance of C_{60}-H in the ^1H-NMR of **1** and **2** to confirm the linkage of C_{60} to PEO once more[8].

In conclusion, a new simple and easy method was further developed for synthesizing C_{60}-containing poly(ethylene oxide) (PEO). It can be expected that many other polymers containing C_{60} moieties could be easily prepared by this new and simple synthetic strategy.

Figure 1 The UV-Vis spectrum of **1** in chloroform



Acknowledgments

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References

1. R. E. Hulfer, J. Conceical, L. P. F. Chibante, *J. Phys. Chem.*, **1990**, *94*, 8634.

2. K. E. Geckeler, A. Hirsch, *J. Am. Chem. Soc.,* **1993**, *115,* 3850.
3. S. H.Friedman, D. L. Decamp, R. P. Sijbesma, *et al., J. Am. Chem. Soc.,* **1993**, *115,* 6506.
4. N. Manolova, I. Rashkov, F. Beguin, H. V. Damme, *Chem. Commun.,* **1993**, 1725.
5. Y. Ederle, C. Mathis, R. Nuffer, *Synth. Met.,* **1997**, *86,* 2287.
6. P. Fang, Ph. D. Thesis, *Wuhan University,* **1999**.
7. Y. Sun, B. Liu, D. K. Moton, *Chem. Commun.,* **1996**, 1699.
8. M. L. Miller, R. West, *Chem. Commun.,* **1999**, 1797.

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