Synthesis of New Biocompatible Multi-Functional Textile Finishing Agent

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1. ABSTRACT

A methacrylate monomer having phospholipid polar group and cell membrane structure is known as highly biocompatible. Based on these properties, new biocompatible multi-functional textile finishing agent was developed using phospholipid copolymer. 2-Methacryloyloxyethyl phosphorylcholine (MPC) was synthesized using 2-hydroxyethyl methacrylate (HEMA), 2-chloro-2-oxo-1,3,2-dioxaphospholane (COP), trimethylamine (TMA) and triethylamine (TEA), and then polymerized to prepare MPC copolymer by radical polymerization using AIBN. The structures of MPC and MPCE were characterized by FTIR and 1H NMR and will be evaluated as textile finishing agent in further study.

2. INTRODUCTION

Environmental technology (ET) is already one of the most important technology area throughout the world and the demand of environmental friendly and biocompatible products are continuously increasing. Also in textile industry, lots of functional eco products such as antimicrobial, deodorization, skin aging and atopy prevention, and moisturizing are continuously developed and introduced. Especially, biocompatible moisture finishing products for skin protection such as squalene, collagen, chitosan, hyaluronic acid, and ceramide are very wide spread in cosmetics or medical care area. However, in textile industry, these materials were not so popular since the performance was unsatisfactory when applied to synthetic fibers.

Phospholipid polymer known as lipidure consists of hydrophilic phosphoric acid parts and hydrophobic lipid parts forming fats. It was already verified that they have functions of moisturizing, anti skin aging, antimicrobial and excellent stability since they have similar structures to cell membrane.

In this study, new biocompatible multi-functional textile finishing agents based on phospholipid MPC copolymer was synthesized and characterized for synthetic fibers such as polyester and nylon.

3. EXPERIMENTAL

2-Methacryloyloxyethyl phosphorylcholine (MPC) was synthesized using 2-hydroxyethyl methacrylate (HEMA), 2-chloro-2-oxo-1,3,2-dioxaphospholane (COP), trimethylamine (TMA) and triethylamine (TEA), and then polymerized to prepare MPC copolymer by radical polymerization using AIBN. Into a 500 ml double walled vessel equipped with constant low temp circulator and dropping funnel, dry THF and dry diethyl ether were placed by solvent, and TEA and HEMA were added to the solution. After the solution was cooled at 0 ℃, COP in dry THF and dry diethyl ether were added dropwise to the stirred solution over a period of 1hr. The temperature of the reaction mixture was maintained at room temperature for 4hr. Then, the precipitate in the reaction mixture which was triethylammonium chloride was filtered off and the filtrate was evaporated under reduced pressure. Dry THF and dry diethyl ether were placed by solvent, and TEA and HEMA were added to the solution. After the solution was cooled at 0 ℃, COP in dry THF and dry diethyl ether were added dropwise to the stirred solution over a period of 1hr. The temperature of the reaction mixture was maintained at room temperature for 4hr. Then, the precipitate in the reaction mixture which was triethylammonium chloride was filtered off and the filtrate was evaporated under reduced pressure. Dry THF were added to the residue to precipitate a small amount of triethylammonium chloride by filtration. By evaporation of filtrate under reduced pressure, colorless liquid intermediate, 2-(2-oxo-1,3,2-dioxaphospholoyloxy)ethyl methacrylate (OPEMA) was obtained.

$^1$H NMR (CDCl$_3$): $\delta$=1.95 (CH$_3$, 3H), 4.10-4.40 (CH$_2$, 8H), 5.60 (CH$\equiv\equiv$, 1H), and 6.15 (CH$\equiv\equiv$, 1H).

OPEMA and dry acetonitrile were placed in a two-necked flask equipped with a thermometer and condenser. After the flask was cooled at -20 ℃, trimethylamine were rapidly added to the solution and the pressure bottle was closed and allowed to warm up to room temperature. After it was heated at 60 ℃ for 24h, the mixture was evaporated under reduced pressure. Dry acetonitrile were added to the residue and evaporated under pressure to give colorless liquid produce, MPC.

In the similar synthetic route, MPCE was synthesized using triethylamine(TEA) instead of trimethylamine(TMA). TEA was added at room temperature because it was more stable than TMA and colorless liquid product, MPCE was obtained.

The synthesis scheme is shown in Scheme 1 and the NMR spectrum is shown in Figure 1.
The desired amounts of MPC, MPCE or their derivatives, and AIBN were dissolved in methanol-THF mixture and then taken into polymerization tubes. After oxygen in the tubes was eliminated by bubbling of Nitrogen into the solution, the tubes were sealed. The reaction mixture was then evaporated under reduced pressure and acetone was added to precipitate and extracted with acetonitrile in Soxhlet for 24h and dried in a vacuum to give MPC copolymer.

4. REFERENCES


Figure 1. 1H NMR (CDCl3)pectrum of OPEMA

Scheme 1. The synthesis of MPC and MPCE