PJ1) Synthesis of β-hydroxyalkylamide by energy-saving microwave

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1. Introduction

Microwave heating is one of electroheat techniques, such as induction, radio frequency, and infra-red heating. In some cases, these processes can replace conventional heating or drying systems because some conventional systems are very bulky, not easy to operate and can pollute the environment due to harmful emissions. The major advantages of using microwaves are rapid heat transfer, selective heating, speed of fast switching on and off, and pollution-free environment (Larhed, 2001; Jung et al., 2001).

One of the most effective crosslinkers of thermosetting powder coatings of carboxyl-terminated polyesters was triglycidyl isocyanurate (TGIC). However, the toxicity of TGIC has led to the use of new crosslinker, such as b-hydroxyalkyl amides, commercialized under the name of Primid. In this study, microwave heating system was applied for the synthesis of β-hydroxyalkylamide. The obtained products were compared to that synthesized from conventional heating system.

2. Experimental

Dimethyl adipate (DMA, Tokyo Chemical Industry Co.), diethanol amine (DEA, KPX Green Chemical), sodium methoxide ($NaOCH_3$, Sigma Aldrich) were used without further purification. Microwave-assisted synthesis was conducted by a microwave reactor (2.45 GHz, 1 KW, Hankook Microwave Co.). DMA(1 mol.) and DEA (2 mol.) were placed in a three-necked flask equipped with a thermometer, a stirrer, a condenser with a drying tube. Sodium methoxide as a catalyst (0.1 wt% of DEA) was added and the mixture was heated to 85°C under stirring (Fig. 1). After 30 min, the methanol in the product was removed in vacuum. Conventional reaction was carried out in heating mantel.

3. 결과 및 고찰

To save energy, β-Hydroxyalkylamide, which is normally produced by oil bath heating, was synthesized by microwave heating. Performances of products obtained by both heating systems were compared by the measurements of thermal property, FT-IR, and flowability. For finishing the reaction, the conventional heating system took 300 min of reaction time whereas the microwave heating system required much short time, 100 min of reaction time. The yields of products synthesized by both heating systems are about 99% and its performance was almost the same. These results suggest that the microwave as a heating system is an effective method for the reaction of β-hydroxyalkylamide.

4. 참고문헌

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