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# Transesterification of Vinyl Acetate With 2-Phenyl-1-Propanol Using Distannoxane Catalyst

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## ABSTRACT

Preliminary reactions involved model compounds to gain a better understanding of reaction mechanisms on both transesterification and Michael addition reactions. So the aim of this research was to investigate the enzyme-catalyzed functionalization of polymers. For this purpose, I have prepared this paper for transesterification of vinyl acetate with 2-phenyl-1-propanol by using the distannoxane catalyst.

Keywords : Transesterification, 2-phenyl-1-propanol, PPOH, distannoxane catalyst, vinyl acetate

## I. INTRODUCTION

MODEL REACTION

Distannoxane-catalyzed Transesterification of Vinyl Acetate with 2-Phenyl-1-propanol.

Bis [dibutylchlorotin (IV)] oxide (distannoxane) was employed as transesterification catalyst as reported in the paper (1). 2-Phenyl-1-propanol (0.7 mL, 5 mmol, 0.5 mol/L) was reacted with vinyl acetate (5 mL, 0.05 mol, 5 mol/L) in THF (5 mL) in the presence of distannoxane (0.1 g, 0.2 mmol, 2 x 10-2 mol/L) at 50 °C. In a separate experiment, the same conditions as the enzymatic transesterification of vinyl acetate with 2-phenyl-1-propanol were employed. Specifically, 2-phenyl-1-propanol (0.3 mL, 2 mmol, 0.1 mol/L) was reacted with vinyl acetate (0.6 mL, 6 mmol, 0.3 mol/L) in hexane (20 mL) in the presence of distannoxane (0.3 mg, 5 x 10-4 mmol, 2 x 10-5 mol/L) at 50 °C. Both reactions were monitored with TLC using ethyl acetate/hexane (1/2, v/v) as eluent.

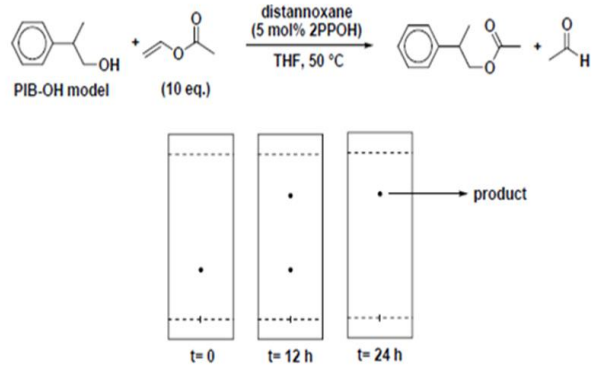


Figure 4.5. TLC monitoring of the transesterification of vinyl acetate with 2PPOH using distannoxane catalyst [eluent: ethyl acetate/hexane (1/2; vol/vol)].

## II. RESULT AND DISCUSSION

Comparison of Chemical and Enzymatic Catalysis: Distannoxane-catalyzed Transesterification of Vinyl Acetate with 2-phenyl-1-propanol (2).

After showing that the use of vinyl ester is necessary for successful polymer functionalization, the catalytic activity of CALB was compared with that of a conventional catalyst in the transesterification (3) of vinyl acetate with the same model compound, 2PPOH. Bis (dibutylchlorotin (IV)) oxide, a commercially available 1,3-disubstituted tetra alkyldistannoxane, was used as the catalyst since distannoxanes are known as highly efficient transesterification catalysts.

Orita et al were able to obtain 98% conversion in the reaction of 2-phenylpropanol (1M) with vinyl acetate (5 M) in THF using bis (dibutylchlorotin(IV)) oxide (0.05 M, 5 mol% of alcohol) as the catalyst. The acylation of 2PPOH (0.47 M) with vinyl acetate (5.07 M) was carried out in THF in the presence of distannoxane (0.02 M, 5 mol% of 2PPOH) at 50 °C. A higher number of equivalents of vinyl acetate was used to ensure complete conversion.

The progress of the reaction was monitored with TLC using ethyl acetate/hexane (1/2; vol/vol) as the eluent mixture. The reaction was incomplete after 12 hours, as the spot at Rf = 0.3 corresponding to 2PPOH could still be observed. This spot disappeared completely after 24 hours of reaction time (4). In comparison, the CALB-catalyzed reaction was complete in 2 hours in hexane. The reaction was also repeated using the same conditions as the enzymatic reaction. Specifically, 2PPOH (0.1 M) was reacted with vinyl acetate (0.3 M) in hexane in the presence of distannoxane (2 x 10-5 M) at 50 °C. However, no progress in the reaction could be detected by TLC even after 24 hours probably due to rather low catalyst concentration. Therefore, CALB is a more reactive transesterification catalyst than distannoxane, and it is an environmentally preferred alternative.

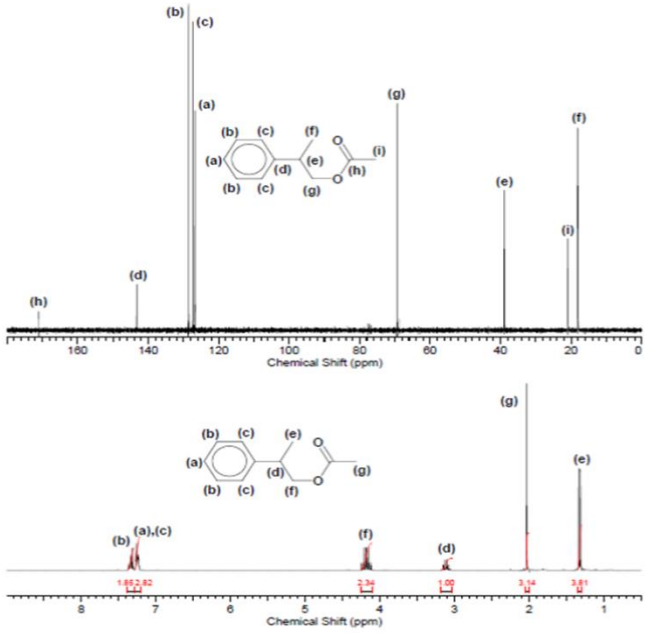


Figure 4.3. NMR spectra of the transesterification product of vinyl acetate with 2PPOH: (top) $^{13}C$ NMR spectrum and (bottom) $^{1}H$ NMR spectrum (solvent: $CDCl_3$).

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