

Electrophilic aromatic arylation polycondensation synthesis of wholly aromatic polyketone composed of 2,2'-dimethoxy-1,1'-binaphthylene moiety

Akiko Okamoto, Ryosuke Mitsui, Katsuya Maeyama, Hiromu Saito, Hideaki Oike, Yoshihiko Murakami, Noriyuki Yonezawa *

Department of Organic and Polymer Materials Chemistry, Tokyo University of Agriculture and Technology, Koganei, Tokyo 184-8588, Japan

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Dedicated to Professor Teiji Tsuruta on the occasion of his 88th birthday (Beiju).

Abstract

A wholly aromatic polyketone containing 2,2'-dimethoxy-1,1'-binaphthylene moiety was successfully synthesized *via* electrophilic aromatic arylation polycondensation with the aid of trifluoromethanesulfonic acid (TfOH) or phosphorus(V) oxide–methanesulfonic acid mixture (P₂O₅–MsOH). The polycondensation reactions employing two sets of monomers of opposite combination that should afford the same structure of repeating unit showed distinct results. The polycondensation employing 2,2'-dimethoxy-1,1'-binaphthyl (**4**) as the acyl-acceptant monomer proceeded to give medium-molecular-weight polymer. The polymer synthesis *via* transformation of biaryl **4** into the corresponding acyl-donor monomer **12** followed by polycondensation with 2,2'-dimethoxybiphenyl (**1**) predominated the procedure of direct usage of biaryl **4** as the acyl-acceptant monomer. Acyl-acceptant monomer has been demonstrated to play a more crucial role in determination of polymerizability than acyl-donor one.

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1. Introduction

Aromatic polyketones are recognized as a sort of aromatic polymers having high thermal stabil-

ity, excellent chemical resistance, and good mechanical strength. Almost all of the aromatic polyketones are classified as aromatic poly(ether ketone)s [1]. The presence of ether linkage greatly facilitates the formation reaction. Nucleophilic aromatic substitution is generally employed as the chain-elongation of this type of polymer since high degree of polymerization is readily achieved

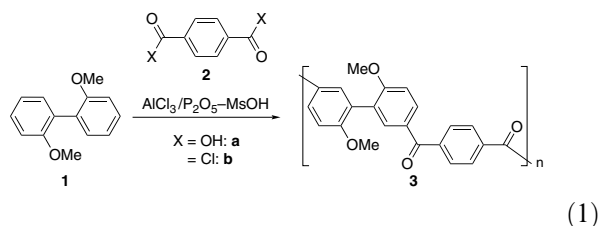
* Corresponding author. Tel.: +81 42 388 7053; fax: +81 42 388 7291.

E-mail address: yonezawa@cc.tuat.ac.jp (N. Yonezawa).

with excellent selectivity and sufficient reactivity [2–6].

On the other hand, there are only a few papers on synthesis of wholly aromatic polyketones, a type of aromatic polyketone having the main chain skeleton composed of only aromatic rings and ketonic carbonyl groups. The preparation method of wholly aromatic polyketones is significantly restricted compared to aromatic poly(ether ketone)s. Electrophilic aromatic arylation seems the most advantageous candidate for the formation of ketonic carbonyl group–aromatic ring bond. However, the introduction of a ketonic carbonyl group reduces the electron density of the aromatic rings resulting in large disturbance against further arylation. Consequently, the corresponding polymerization hardly maintains high reactivity, good selectivity, and sufficient consecutiveness. Actually, transition metal complex-mediated aryl–aryl coupling has been the choice of synthetic method for this type of polyketone [7–10].

During the course of the synthetic study of wholly aromatic polyketones [11–25], some kinds of acyl-acceptant arenes have proved to afford this type of polyketone satisfactorily *via* electrophilic aromatic arylation. One of the most effective acyl-acceptant monomers is biaryl **1**. In the electrophilic aromatic arylation, biaryl **1** undergoes double regioselective arylation reactions at the 5,5'-positions successively. It has been revealed that biaryl **1** gives high-molecular-weight wholly aromatic polyketone through Friedel–Crafts arylation polymerization (Eq. (1)) [19,20].

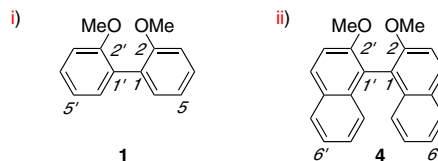


When biaryl **1** was allowed to react with an equimolar amount of 4-chlorobenzoic acid, an approximately statistical mixture was obtained. This result suggests that there is almost no difference in susceptibility to electrophilic aromatic arylation reaction between biaryl **1** and its monoarylated derivative. The constancy of the reactivity before and after the first arylation is

plausibly attributable to the electron-donating methoxy groups and also to the twisted biphenylene unit. The weakened conjugation of the twisted biphenylene moiety might interfere the transfer of the electron-withdrawing effect of the acyl group to the other aromatic ring. In addition, the conjugation of the resulting polyketones is estimated to remain in the satisfactory level to perform the high thermal stability.

The consecutive acyl-accepting ability of the modified 2,2'-dioxybiphenyl and the relating structures has been demonstrated [21–25].

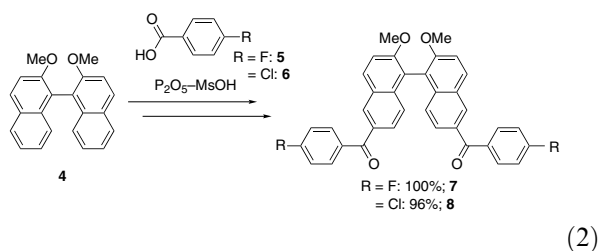
By the way, 2,2'-dimethoxy-1,1'-binaphthyl (**4**) has the essentially identical electronic structure with biaryl **1** (Formulae). Biaryl **4** is one of the derivatives of 1,1'-binaphthol, which is widely employed chiral building blocks.



Formulae. Acyl-acceptant aromatic compound and the analogue.

i) 2,2'-dimethoxybiphenyl (**1**), ii) 2,2'-dimethoxy-1,1'-binaphthyl (**4**).

Recently, the regioselective arylation of biaryl **4** at the 6,6'-positions by phosphorus(V) oxide–methanesulfonic acid mixture (P_2O_5 –MsOH) [26], a direct condensation reagent, was performed (Eq. (2)) [27].



Utilizing such reaction behavior, the syntheses of optically active aromatic poly(ether ketone)s *via* nucleophilic aromatic substitution polycondensation of 6,6'-bis(4-fluorobenzoylated) compound (*S*)-**7** with diphenols [28,29], and that of optically active wholly aromatic polyketone *via* nickel complex-mediated aromatic coupling polycondensation of 6,6'-bis(4-chlorobenzoylated) compound (*S*)-**8** have been achieved [30], respectively (Eq. (3)).

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