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Microwave-assisted regioselective alkylation of naphthalene compounds using alcohols and zeolite catalysts

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ABSTRACT

Regioselective alkylation of naphthalene compounds with alcohols smoothly proceeded in the presence of zeolite catalysts under microwave irradiation. A H-mordenite (H-M) zeolite catalyst (SiO_2/Al_2O_3 ratio=240) showed the highest efficiency. In the microwave reactions, high reaction rates and high selectivities for 2,6-dialkylnaphthalenes were achieved. In the best case for the reaction of 2isopropylnaphthalene with isopropyl alcohol, the conversion and the selectivity were 43.5% and 66.4%, respectively. In di-*tert*-butylation of naphthalene with *tert*-butyl alcohol, the conversion and the selectivity reached 86.5% and 70.4%, respectively. The conversions and the selectivities were generally higher than those obtained by conventional oil bath heating.

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1. Introduction

2,6-Dialkylnaphthalenes are useful aromatic compounds to prepare various functional materials. For instance, some 2,6dialkylnaphtahlenes can be converted into naphthalene-2,6dicarboxylic acid (NDCA). NDCA can be used as a monomer for poly(ethylene-2,6-naphthalenedicarboxylate)(PEN), which has wide applicability as mechanically strong and thermally stable films, packaging materials, coatings, adhesives, etc. [1,2]. To synthesize 2,6-dialkylnaphthalenes, Friedel-Crafts type regioselective alkylations of naphthalene compounds using zeolite catalysts and sterically large alcohols such as isopropyl alcohol [3-10] and tert-butyl alcohol [11-17] have been investigated to date. However, it still seems not so easy to achieve high reaction rates and/or high selectivities for 2,6-dialkylnaphtahlenes. On the other hand, microwave irradiation has been effectively utilized in recent organic synthesis hoping for improvement of the reaction rates and/or the selectivities for desired products [18-20]. Alcohol compounds with hydroxyl groups have relatively large dielectric loss factors and are considered to be good materials for microwave absorption (vide infra). Therefore, we have examined zeolite-catalyzed alkylations of naphthalene compounds using alcohols as alkylating agents. As the results, we have found that alkylation of naphthalene compounds (2-isopropylnaphthalene, naphthalene) using a suitable zeolite catalyst and some alcohols (isopropyl alcohol, *tert*-butyl alcohol) smoothly proceeds under microwave irradiation to give the corresponding 2,6-dialkylnaphtahlenes in reasonably high selectivities.

2. Experimental

2.1. Reagents and instruments

Liquid compounds such as 2-isopropylnaphthalene (2-1a), isopropyl alcohol (2a), *tert*-butyl alcohol (2b), decahydronaphthalene (DHN), and 1,2-dichlorobenzene (DCB) were used after drying with molecular sieve 4A. Naphthalene (1b) was purchased and used as it was. An activated carbon was obtained from Wako Pure Chem. Ind., Ltd., and used after heating at 120 °C for 2 h. Authentic samples of 1-isopropylnaphthalene (1-1a), 2,6- and 2,7-diisopropylnaphthalene (2,6- and 2,7-3a, respectively), and 2,6-and 2,7-di-*tert*-butyl-naphthalene (2,6- and 2,7-3b, respectively) were purchased from Tokyo Chem. Ind. Co., Ltd. An authentic sample of 2-*tert*-butyl-naphthalene (2-1c) was prepared according to a literature method [21], and its ¹H and ¹³C NMR chemical shifts were in good agreement with the reported values [22]. Zeolite catalysts were the commercial products of Tosoh Co., Ltd. Their

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Table 1

Properties of commercial zeolites used in the present alkylation reaction.

Zeolite	Product code ^a	SiO ₂ /Al ₂ O ₃	BET surface area (m ² /g)	Na ₂ O (%)	LOI ^b (%)
H-beta (27)	930HOA	27	670	<0.01	4.7
H-Y (14)	360HUA	14	550	0.02	0.5
H-M (15)	620HOA	15	390	0.45	11.2
H-M (33)	660HOA	33	400	0.03	12.2
H-M (240)	690HOA	240	450	<0.05	5.5

^a All the zeolites were obtained from Tosoh Co., Ltd.

^b Loss on ignition.

Table 2

Reactions of 2- 1a with 2a ª
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Run	Catalyst ^b	Additive	Solvent ^c	Method	Temp. (°C)	Time (min)	Yield or recovery ^d (%)						2,6- 3a /2,7- 3a	Conv. of 1a ^e (%)	Select. for
							1b	1 -1a	2 -1a	2,6- 3a	2,7- 3a	Others			2,0 -3d [•] (%)
1	H-beta (27)	AC	DHN	MW	225	30	7.8	4.0	45.4	19.8	20.0	3.0	1.0	50.6	39.1
2	H-Y(14)	AC	DHN	MW	225	30	6.1	2.3	25.3	19.0	19.0	28.3	1.0	72.4	26.2
3	H-M (16)	AC	DHN	MW	225	30	< 0.1	2.1	87.7	1.0	0.4	8.8	2.5	10.2	9.8
4	H-M (33)	AC	DHN	MW	225	30	0.2	2.5	76.7	8.9	3.4	8.3	2.6	20.8	42.8
5	H-M (240)	AC	DHN	MW	225	30	3.6	4.6	64.4	19.6	7.8	1.9	2.5	31.0	63.2
6	H-M (240)	AC	DHN	MW	175	30	< 0.1	2.0	83.1	7.6	3.1	4.2	2.5	14.9	51.4
7	H-M (240)	AC	DHN	MW	200	30	1.7	2.3	68.7	19.5	7.3	2.2	2.7	29.0	67.4
8	H-M (240)	AC	DHN	MW	250	30	5.5	5.5	51.7	20.0	11.8	7.3	1.7	42.7	46.9
9	H-M (240)	AC	DHN	MW	275	30	< 0.1	4.0	44.3	23.3	14.0	8.9	1.7	51.7	45.1
10	H-M (240)	AC	DHN	MW	200	15	0.4	2.7	69.6	17.1	7.0	3.2	2.4	27.4	62.4
11	H-M (240)	AC	DHN	MW	200	60	0.2	3.0	65.8	21.6	8.9	0.5	2.4	31.2	69.2
12	H-M (240)	AC	DHN	OB	200	15	< 0.1	2.1	82.6	9.7	4.1	1.5	2.4	15.3	63.4
13	H-M (240)	AC	DHN	OB	200	30	< 0.1	2.1	81.6	10.4	4.2	1.7	2.5	16.3	63.8
14	H-M (240)	AC	DHN	OB	200	60	< 0.1	0.7	81.3	11.3	4.6	2.1	2.5	18.0	62.7
15	H-M (240)	-	DCB	MW	200	30	0.4	2.1	54.4	28.9	10.4	3.8	2.8	43.5	66.4

^a General reaction conditions: **1a** (2-**1a**:1-**1a** = 97.8:2.2) 1.5 mmol, **2a** 1.5 mmol, catalyst 100 mg, activated carbon (AC) 50 mg, solvent 0.5 mL, microwave (MW) or oil bath (OB).

The products were analyzed with a GC instrument of Shimadzu GC-

17A with a capillary column of Zebron ZB-50 (0.25 mm i.d. \times 30 m)

using an internal standard (tetradecane). Complex dielectric con-

stants of liquid compounds were measured by a perturbation

method [23] with a system of Kanto Electronic Application and

Development Inc. comprising of a vector network analyzer of Agi-

lent 8720ES and a 2.45 GHz cylindrical cavity of TM₀₂₀ mode using

Teflon-type sample tubes. ¹H and ¹³C NMR spectra were measured

by a Jeol JNM-LA600 instrument using chloroform-d solvent.

^b Figures in parentheses are the ratios of SiO_2/Al_2O_3 .

^c DHN = decahydronaphthalene, DCB = 1,2-dichlorobenzene.

^d Yield and recovery were estimated by GC.

^e Conversion of **1a** (%) = 100 – recovery of **1** (1-**1a**+2-**1a**).

 $^{\rm f}$ Selectivity for 2,6-**3a** (%)=100 \times yield of 2,6-**3a**/conversion of **1a**.

properties are summarized in Table 1. These zeolite catalysts were used after heating at 500 °C for 5 h.

Microwave irradiation experiments were carried out with a CEM Discover instrument (single mode type, microwave max power 300 W) using closed Pyrex glass vials (ca. 10 mL) with Teflon-coated septa. The reaction temperatures were monitored by a radiation thermometer attached to the microwave instrument. The temperatures measured by the radiation thermometer were calibrated by a fiber optic thermometer and/or standard mercury thermometers.

Table 3

Reactions of 1b with 2b^a.

Run	2b (equiv.)	Catalyst ^b	Method	Temp. (°C)	Time (min)	Yield or recovery ^c (%)						2,6- 3b /2,7- 3b	Conv. of 1b ^d (%)	Select. for
						1b	1 -1c	2 -1c	2,6- 3b	2,7- 3b	Others			2,0- 30 ° (%)
1	2.0	H-M (240)	MW	150	30	67.8	<0.1	19.2	9.8	1.2	2.0	8.2	32.2	30.4
2	2.0	H-M (240)	MW	160	30	33.1	0.4	25.7	33.6	1.9	5.3	17.7	66.9	50.3
3	2.0	H-M (240)	MW	175	30	29.7	0.6	25.7	37.7	1.6	4.7	23.6	70.3	53.6
4	2.0	H-M (240)	MW	200	30	19.6	0.7	29.5	49.3	0.6	0.3	82.2	80.4	61.4
5	2.0	H-M (240)	MW	225	30	15.4	0.9	36.3	42.2	0.4	4.8	106	84.6	49.9
6	2.0	H-M (240)	MW	175	5	65.9	0.1	20.5	11.8	0.7	1.0	16.9	34.1	34.6
7	2.0	H-M (240)	MW	175	10	39.0	0.2	26.5	30.8	1.0	2.5	30.9	61.0	50.4
8	2.0	H-M (240)	MW	175	60	20.3	0.7	24.5	47.2	1.3	6.0	36.3	79.7	59.3
9	1.0	H-M (240)	MW	200	30	36.7	0.8	41.2	18.4	1.0	1.9	18.4	63.3	29.1
10	3.0	H-M (240)	MW	200	30	9.3	0.8	21.7	61.3	0.7	6.2	87.6	90.7	67.5
11	3.5	H-M (240)	MW	200	30	13.5	0.5	18.8	60.9	1.0	5.3	60.9	86.5	70.4
12	2.0	H-M (240)	OB	175	30	73.5	< 0.1	14.6	9.2	0.8	1.9	11.5	26.5	34.9

^a General reaction conditions: **1b** 1.5 mmol, **2b** 1.5–5.25 mmol, catalyst 100 mg, activated carbon (AC) 50 mg, decahydronaphthalene (DHN) 0.5 mL, microwave (MW) or oil bath (OB).

 $^{\rm b}\,$ Figures in parentheses are the ratios of SiO_2/Al_2O_3.

^c Yield and recovery were estimated by GC.

 $^{\rm d}\,$ Conversion of $1b\,(\%)$ = 100 $-\,$ recovery of 1b.

 $^{e}~$ Selectivity for 2,6-3b (%) = 100 \times yield of 2,6-3b/conversion of 1b.



Fig. 1. A monitoring profile for the reaction of 1b with 2b (200 °C, 30 min).

2.2. Alkylation of naphthalenes

A typical procedure for a microwave reaction was as follows. A mixture of a naphthalene compound **1** (1.5 mmol), an alcohol **2** (1.5–5.25 mmol), a zeolite catalyst (100 mg), activated carbon (50 mg), decahydronaphthalene (DHN, 0.50 mL), tetradecane (internal standard, 50 mg), and a magnetic bar was sealed in a Pyrex glass vial (ca. 10 mL). The mixture was heated by microwave irradiation (microwave max power 300 W) with magnetically stirring under the specified conditions shown in Tables 2 and 3. Identification of the products and estimation of the conversions of **1** and the yields of the products were made by GC and/or GC–MS analyses.

In the microwave irradiation experiment, the irradiation power was automatically adjusted so as to keep the reaction temperature. A typical profile of the changes in the temperature and the irradiation power is shown in Fig. 1 for the reaction of **1b** with **2b** at 200 °C for 30 min.

In an oil bath reaction also, a Pyrex glass vial with the same shape as that of the microwave reaction was used. The glass vial containing starting materials, a solvent, and a catalyst was immersed into an oil bath preheated at the specified temperature, and the heating was continued with magnetic stirring under the conditions shown in Tables 2 and 3.

3. Results and discussion

3.1. Dielectric properties of alcohols

In the microwave-assisted reactions, it seems wise to use starting materials which absorb microwave energy efficiently. For absorption of microwaves, the dielectric loss factor of the material is an important key parameter; the calorific power (P) by microwave absorption is postulated to be proportional to the dielectric loss factor (ε''); $P = 2\pi f \varepsilon_0 \varepsilon'' E^2$, f: microwave frequency, ε_0 : dielectric constant of vacuum, E: intensity of electric field [24]. Therefore, compounds with large dielectric loss factors are considered to be favorable for absorption of microwave energy.

In our study to evaluate dielectric properties of chemical materials by a perturbation method using a 2.45 GHz cylindrical cavity, alcohol compounds tended to have relatively large dielectric loss factors and would become suitable starting compounds for microwave-assisted reactions. Therefore, we have investigated alkylation of naphthalene compounds using alcohols as alkylating agents, hoping for promotion of the reaction by efficient activation of the alcohols by microwave irradiation. As the alcohols, isopropyl alcohol and *tert*-butyl alcohol have been selected, since they have large dielectric loss factors (2.92 and 1.32, respectively), which are much larger than those of non-alcoholic compounds such as hexane and toluene (0.0008 and 0.012, respectively) [25]. In addition, the steric bulkiness of these alcohols is considered to be beneficial for regioselective alkylation.

3.2. Isopropylation of 1a

First, the reaction of 2-isopropylnaphthalene (2-1a) with isopropyl alcohol (2a, 1 equiv.) was examined using several zeolite catalysts (Scheme 1, Table 2). To undergo the reaction smoothly, we added activated carbon (AC), which also absorbed microwave and heated the reaction system, to the reaction system as a susceptor material. In this reaction, 2,6-diisopropylnaphthalene (2,6-3a) and its 2,7-isomer (2,7-3a) were formed as the major products along with a coproduction of water. As minor products, naphthalene (1b), 1-isopropylnaphthalene (1-1a), and other polyalkylated products were also found in the reaction mixture. Small amounts of 1-1a were existing in the starting material 2-1a (2-1a:1-1a=97.8:2.2), but increased amounts of 1-1a were formed in some reactions. Compounds 1b and 1-1a would be formed by dealkylation of 2-1a and alkylation of 1b at the C1 carbon.

A survey of the catalyst efficiency revealed that the values of the conversions and the selectivities for 2,6-**3a** (100 × yield of 2,6-**3a**/conversion of **1a**) were respectively 50.6% and 39.1% for H-beta (SiO₂/Al₂O₃ = 27), 72.4% and 26.2% for H-Y (14), 10.2% and 9.8% for H-M (16), and 20.8% and 42.8% for H-M (33) catalysts (Runs 1–4, Fig. 2). The ratios of 2,6-**3a**/2,7-**3a** were 1.0 for H-beta (27), 1.0 for H-Y (14), 2.5 for H-M (16), and 2.6 for H-M (33). These results showed that mordenite zeolites gave the best result in a viewpoint of the selectivity for 2,6-**3a** among H-Y, H-beta, and H-M catalysts with similar SiO₂/Al₂O₃ ratios. In the H-M zeolite series, the use of zeolite with the SiO₂/Al₂O₃ ratio of 240 gave the best result in both the conversion and the selectivity for **3a**; the values were respectively 10.2% and 9.8% for H-M (16), 20.8% and 42.8% for H-M (33), and 31.0% and 63.2% for H-M (240) (Runs 3–5). High selectivity



Scheme 1.



Fig. 2. Comparison of catalyst efficiency in the reactions of 1a with 2a (200 $^\circ\text{C},$ 30 min).



Fig. 3. Effect of temperature on the reaction of 1a with 2a for 30 min.

for 2,6-dialkylnaphthalene by mordenite catalysts was previously reported by Sugi and co-workers in the reaction of naphthalene with propene [26].

Concerning the reaction temperature, about 200 °C appeared to provide the highest selectivity for **3a** (67.4%) in the range of 175–275 °C, while the conversion of **1a** increased nearly linearly from 14.9% to 51.7% with raising the temperature (Runs 5–9, Fig. 3). On the other hand, the conversion of **1a** and the yield of 2,6-**3a** were respectively 27.4% and 17.1% (15 min), 29.0% and 19.5% (30 min), and 31.2% and 21.6% (60 min) (Runs 7, 10, and 11, Fig. 4). The reaction using oil bath heating in place of microwave irradiation caused a significant decrease in the reaction rate. The time course of the reaction by oil bath heating showed that the conversion and the yield of 2,6-**3a** were respectively 15.3% and 9.7% (15 min), 16.3% and 10.4% (30 min), and 18.0% and 11.3% (60 min) (Runs 12–14, Figs. 4 and 5). Thus the conversions and the yields in the microwave reactions were better than the corresponding oil bath reactions, although the difference in selectivity was small.



Fig. 4. Time courses of the conversion of **1a** and the yield of 2,6-**3a** in the microwave and oil bath reactions (200 °C): (●) conversion of **1a** (MW), (■) yield of 2,6-**3a** (MW), (○) conversion of **1a** (OB), and (□) yield of 2,6-**3a** (OB).



Fig. 5. Comparison between microwave (MW) and oil bath (OB) reactions (200 $^\circ\text{C}$, 30 min).

As an effective solvent, 1,2-dichlorobenzene (DCB) could be used in place of DHN. DCB has much larger dielectric loss factor (2.30) than DHN (0.001) [25] and is likely to act as a susceptor material. When DCB was used, addition of the activated carbon was not needed. The reaction in DCB gave **3a** with a better conversion of **1a** and almost the same selectivity for **3a**; the conversions and the selectivities in the reactions at 200 °C for 30 min were respectively 29.0% and 67.4% in DHN (Run 7) and 43.5% and 66.4% in DCB (Run 15).

3.3. tert-Butylation of 1b

Since the H-M (240) zeolite was most efficient in the abovementioned reaction, dialkylation of naphthalene (1b) by sterically bulky alcohols was investigated using the same H-M catalyst. As the result, tert-butyl alcohol (2b, 2 equiv.) was found to give both high conversion of 1b and high selectivity for 2,6-di-tertbutyInaphthalene (2,6-3b) (Scheme 1, Table 3). Examination of the effect of the reaction temperature disclosed that the conversion tended to increase from 32.2% at 150 °C to 84.6% at 225 °C, while the selectivity for 2,6-3b ($100 \times$ yield of 2,6-3b/conversion of 1b) showed the highest value (61.4%) at about 200 °C in the range of 150-225 °C (Runs 1-5, Fig. 6). The ratios of 2.6-**3b**/2.7-**3b** increased with raising the temperature from 150 to 225 °C, although the temperature of 200 °C was the best in terms of the selectivity for 2.6-3b: the ratios of 2,6-3b/2,7-3b were 8.2, 17.7, 23.6, 82.2, and 106 for 150, 160, 175, 200, and 225 °C, respectively. On the other hand, the time course of the reaction at 175 °C showed that both the conversion and the selectivity increased by prolonging the time from 5 to 60 min, affording 79.7% conversion with 59.3% selectivity for 2,6-3b (Runs 3 and 6-8, Fig. 7).

The ratio of **2b/1b** was also an important factor. Increasing the ratio of **2b/1b** from 1.0 to 3.5 improved both the conversion of **1b** and the selectivity for 2,6-**3b**; the conversions and the selectivities were respectively 63.3% and 29.1% (**2b/1b** = 1.0), 80.4% and 61.4% (2.0), 90.7% and 67.5% (3.0), and 86.5% and 70.4% (3.5) (Runs 4 and 9–11, Fig. 8). Consequently, when the ratio was 3.5, the best result was obtained, providing 86.5% conversion of **1b** and



Fig. 6. Effect of temperature on the reaction of 1b with 2b for 30 min.



Fig. 7. Time courses of the conversion of 1b and the selectivity for 2,6-3b (175 °C).



Fig. 8. Effect of the ratio of 2b/1b on the reaction of 1b with 2b (200 °C, 30 min).



Fig. 9. Comparison between microwave (MW) and oil bath (OB) reactions (175 $^\circ\text{C}$, 30 min).

70.4% selectivity for 2,6-**3b** (Run 11). These values were higher than those reported by Smith et al., 79% and 62%, respectively, in the H-M catalyzed reaction at 180 °C for 18 h by conventional heating [17].

In the reaction of **1b** with **2b** also, when oil bath heating was used in place of microwave irradiation, the conversion of **1b** and the selectivity for 2,6-**3b** profoundly decreased from 70.3% and 53.6% to 26.5% and 34.9%, respectively, in the reaction at 175 °C for 30 min (Runs 3 and 12, Fig. 9).

As an effect of microwave irradiation, microwaves will heat polar alcohols with relatively high dielectric loss factors efficiently to promote the Friedel–Crafts type alkylation reaction. In addition, the zeolite catalyst center has a protic property and seems to be activated by microwave activation, leading to acceleration of the reaction. Furthermore, concomitantly produced water has also a large dielectric loss factor (10.1), and microwave irradiation may cause activation of the molecule with rapid increase of the temperature, resulting in smooth desorption of the water molecule from the zeolite catalyst. These possible effects of the microwave irradiation would promote the reaction, although further study is required to elucidate the mechanism of the present microwave-assisted reaction more clearly.

4. Conclusion

Regioselective mono- or dialkylation of naphthalene compounds with alcohols (isopropyl alcohol, *tert*-butyl alcohol) were achieved by combined use of microwave irradiation and a H-M catalyst with the ratio of SiO₂/Al₂O₃ being 240. The microwaveassisted reaction tended to provide higher conversions of the starting naphthalene compounds and better selectivities for the desired 2,6-dialkylnaphthalene products than the conventional reaction by oil bath heating. Further study on the development of efficient chemical processes using microwave irradiation and zeolite catalysts is under way.

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