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ORGANIC CHEMISTRY | RESEARCH ARTICLE

P_2O_5 supported on SiO_2 as an efficient and reusable catalyst for rapid one-pot synthesis of carbamatoalkyl naphthols under solvent-free conditions

Atefeh Ghasemi¹, Abolghasem Davoodnia^{1*}, Mehdi Pordel¹ and Niloofar Tavakoli-Hoseini¹

Abstract: Under mild conditions and without any additional organic solvent, synthesis of carbamatoalkyl naphthols by the one-pot three-component reaction of β -naphthol with a wide range of aromatic aldehydes and methyl carbamate could be carried out in the presence of P_2O_5 supported on SiO_2 (P_2O_5/SiO_2). The results showed that the catalyst has high activity and the desired products were obtained in high yields in short reaction times. Other beneficial features of this protocol include inexpensive and easily obtained catalyst, simple work-up, and the recyclability and reusability of the catalyst for up to five consecutive runs.

Subjects: Organic Chemistry; Environmental Chemistry; Inorganic Chemistry

Keywords: P_2O_5/SiO_2 ; carbamatoalkyl naphthols; solvent-free conditions

1. Introduction

Multi-component reactions (MCRs) have attracted much interest and are highly regarded in medicinal chemistry and discovery and synthesis of natural products because they are one-pot processes that bring together three or more components and show high atom economy and high selectivity (Brauch, van Berkel, & Westermann, 2013; Dömling, 2006; Slobbe, Ruijter, & Orru, 2012; Thompson, 2000; Touré & Hall, 2009). They consist of two or more steps which are carried out without isolation of any intermediate. They also provide a rapid and efficient approach to organic synthesis (Davoodnia, Tavakoli-Nishaburi, & Tavakoli-Hoseini, 2011; Gholipour, Davoodnia, & Nakhaei-Moghaddam, 2015; Meerakrishna, Periyaraja, & Shanmugam, 2016). Still, great efforts are being made to develop new



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Abolghasem Davoodnia was born in 1971, Mashhad, Iran. He studied chemistry at Tehran University, Tehran, Iran, where he received BSc in 1994. He received his MSc degree in organic chemistry in 1997 from Ferdowsi University of Mashhad, Mashhad, Iran, under the supervision of Professor Majid M. Heravi and completed his PhD in organic chemistry in 2002 under the supervision of Prof. Mehdi Bakavoli at the same university. Currently, he is working as a professor at the Chemistry Department, Mashhad Branch, Islamic Azad University, Mashhad, Iran. He has published over 140 peer-reviewed articles in ISI journals. His current research interest is on heterocyclic chemistry, catalysis and new synthetic methodologies.

PUBLIC INTEREST STATEMENT

Synthesis of carbamatoalkyl naphthols which can be converted to important biologically active 1-aminomethyl-2-naphthol derivatives by carbamate hydrolysis is of great interest. Therefore, in this paper, a simple and efficient method for the synthesis of these compounds by the one-pot three-component reaction of β -naphthol, aromatic aldehydes, and methyl carbamate using P_2O_5/SiO_2 as catalyst has been reported. The method was fast and the desired products were obtained within a few minutes in high yields under solvent-free conditions. Other advantages of this protocol include inexpensive and easily obtained catalyst, simple work-up, and the recyclability and reusability of the catalyst.

MCRs and improve known ones such as the synthesis of carbamatoalkyl naphthols. These compounds can be converted to important biologically active 1-aminomethyl-2-naphthol derivatives by carbamate hydrolysis. The hypotensive and bradycardiac effects of later compounds have been evaluated (Dingermann, Steinhilber, & Folkers, 2004; Shen, Tsai, & Chen, 1999). A literature survey revealed that a number of methods were known about the synthesis of carbamatoalkyl naphthols via the one-pot three-component reaction of β -naphthol, an aldehyde, and a carbamate in the presence of a variety of catalysts such as cerium ammonium nitrate (CAN) (Wang, Liu, Song, & Zhao, 2013), $\text{Mg}(\text{HSO}_4)_2$ (Ghashang, 2014), Zwitterionic salt (Kundu, Majee, & Hajra, 2010), Tween® 20 (Yang, Jiang, Dong, & Fang, 2013), ionic liquids (Shaterian & Hosseinian, 2014; Zare, Yousofi, & Moosavi-Zare, 2012), PPA-SiO₂ (Shaterian, Hosseinian, & Ghashang, 2009), $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (Song, Liu, Sun, & Cui, 2014), aluminum methanesulfonate ($\text{Al}(\text{MS})_3 \cdot 4\text{H}_2\text{O}$) (Song, Sun, Liu, & Cui, 2013), $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ (Wang, Wang, Zhao, & Wan, 2013), and $\text{Mg}(\text{OCCF}_3)_2$ (Mohammad Shafiee, Moloudi, & Ghashang, 2011). Though each of these methods has its own advantage, the discovery of new and efficient catalysts with high catalytic activity, short reaction times, recyclability, and simple reaction work-up for the preparation of carbamatoalkyl naphthols is of great interest.

Phosphorus pentoxide supported on silica gel ($\text{P}_2\text{O}_5/\text{SiO}_2$) has received considerable attention as an efficient, heterogeneous, eco-friendly, highly reactive, stable, easy to handle, and non-toxic catalyst for various organic transformations including acetalization of carbonyl compounds (Mirjalili, Zolfigol, Bamoniri, Amrollahi, & Hazar, 2004), sulfonylation and nitration of aromatic compounds (Hajipour & Ruoho, 2005; Hajipour et al., 2005), *N*-acylation of sulfonamides (Massah et al., 2009), the Ritter and Schmidt Reactions (Eshghi & Hassankhani, 2006; Tamaddon, Khoobi, & Keshavarz, 2007), Fries and Beckmann rearrangement (Eshghi & Gordi, 2003; Eshghi, Rafie, Gordi, & Bohloli, 2003), the cross-aldol condensation (Hasaninejad, Zare, Balooty, Mehregan, & Shekouhy, 2010), and also the preparation of bis(indolyl)methanes (Hasaninejad, Zare, Sharghi, Niknam, & Shekouhy, 2007), highly substituted imidazoles (Shaterian, Ranjbar, & Azizi, 2011), 4,4'-epoxydicoumarins (Wu & Wang, 2011), Schiff bases (Naeimi, Sharghi, Salimi, & Rabiei, 2008), 1-substituted 1*H*-1,2,3,4-tetrazoles (Habibi, Nasrollahzadeh, Mehrabi, & Mostafaei, 2013), and β -enaminones (Mohammadzadeh, Hasaninejad, Bahramzadeh, & Sardari Khanjarlou, 2009), affording the corresponding products in excellent yields and high selectivity. Other applications of $\text{P}_2\text{O}_5/\text{SiO}_2$ in organic synthesis have been reviewed by Eshghi and Hassankhani (2012).

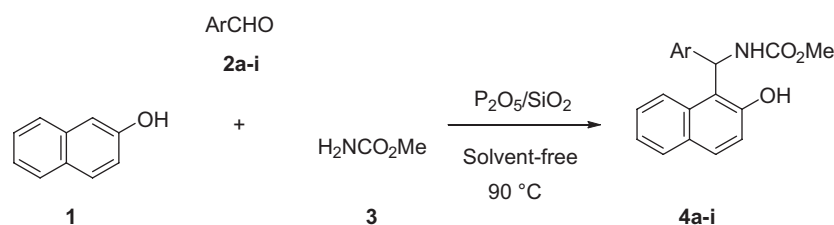
On the other hand, in recent years, considerable interest has been devoted to finding new methodologies for the synthesis of organic compounds in solvent-free condition (Davoodnia, Basafa, & Tavakoli-Hoseini, 2016; Kumar et al., 2016). The toxicity and volatile nature of many organic solvents have posed a serious threat to the environment. Thus, design of solvent-free catalytic reaction has received tremendous attention in recent times in the area of green synthesis (Bettanin, Botteselle, Godoi, & Braga, 2014).

Considering the above facts and also in extension of our previous studies on the development of new environmental friendly methodologies in the synthesis of organic compounds using reusable catalysts (Davoodnia, Allameh, Fazli, & Tavakoli-Hoseini, 2011; Davoodnia, Khashi, & Tavakoli-Hoseini, 2013; Davoodnia, Zare-Bidaki, & Behmadi, 2012; Dehghan, Davoodnia, Bozorgmehr, & Bamoharram, 2016; Khashi, Davoodnia, & Prasada Rao Lingam, 2015; Moghaddas, Davoodnia, Heravi, & Tavakoli-Hoseini, 2012; Nakhaei & Davoodnia, 2014; Nakhaei, Yadegarian, & Davoodnia, 2016; Taghavi-Khorasani & Davoodnia, 2015), we report here the first application of $\text{P}_2\text{O}_5/\text{SiO}_2$ as an efficient, low cost and reusable catalyst for the efficient solvent-free synthesis of carbamatoalkyl naphthols by the one-pot three-component reaction of β -naphthol (**1**) with aromatic aldehydes (**2a-i**) and methyl carbamate (**3**) (Scheme 1).

2. Results and discussion

To begin our study $\text{P}_2\text{O}_5/\text{SiO}_2$ was prepared according to the method reported by Eshghi (Eshghi, Rafei, & Karimi, 2001). Grinding of the mixture of P_2O_5 and SiO_2 in dry conditions for 30 min gave the $\text{P}_2\text{O}_5/\text{SiO}_2$ reagent as white powder. As shown in Scheme 2, the hydroxyl groups in silica gel can be

Scheme 1. P_2O_5/SiO_2 catalyzed synthesis of carbamatoalkyl naphthols.



phosphorylated to give the relatively stable P_2O_5/SiO_2 . This reagent that can act as an acid catalyst has less sensitivity to moisture than P_2O_5 (Eshghi & Hassankhani, 2012).

Different reaction parameters were optimized for the synthesis of compound **4c** by the one-pot three-component reaction of β -naphthol (**1**) (1 mmol), 4-chlorobenzaldehyde (**2c**) (1 mmol), and methyl carbamate (**3**) (1.1 mmol) as a model reaction in the absence and presence of P_2O_5/SiO_2 as catalyst. The results are summarized in Table 1. Only trace amounts of the product **4c** was formed in the absence of the catalyst in refluxing H_2O or EtOH and also under solvent-free conditions (Entries 1–3) indicating that the catalyst is necessary for the reaction. Several reactions were scrutinized using various solvents, such as H_2O , MeOH, EtOH, CH_3CN , CH_2Cl_2 , and also under solvent-free conditions in the presence of P_2O_5/SiO_2 as catalyst. As shown in Table 1, the trial reaction gives the best yield in the presence of 0.05 g of P_2O_5/SiO_2 under solvent-free conditions and proceeds smoothly at 90°C to afford the desired product **4c** in 2 min (entry 14). With tested solvents, the reaction gave low yields within comparable reaction times. Therefore, 0.05 g of the catalyst P_2O_5/SiO_2 under solvent-free condition at 90°C were found to be the optimized conditions. All subsequent reactions were carried out in these optimized conditions.

Under the optimized reaction conditions, we investigated the scope and the limitations of the reaction employing a variety of aromatic aldehydes. The results are summarized in Table 2. Almost all reactions worked well and the desired compounds were obtained in high yields within short reaction time. Under the same conditions however, low yields of the products were obtained using aliphatic aldehydes.

Because of importance of recyclability and reusability of catalysts in organic reactions, the recovery and catalytic activity of recycled P_2O_5/SiO_2 was explored. For this purpose, the synthesis of compound **4c** was again studied under optimized conditions. The P_2O_5/SiO_2 catalyst was readily recovered from the reaction mixture using the procedure outlined in the experimental section. The separated catalyst was washed with hot ethanol and then dried at 50°C under vacuum for 1 h before being reused in a similar reaction. The catalyst could be used at least four times with only a slight reduction in activity

Scheme 2. Preparation of P_2O_5/SiO_2 .

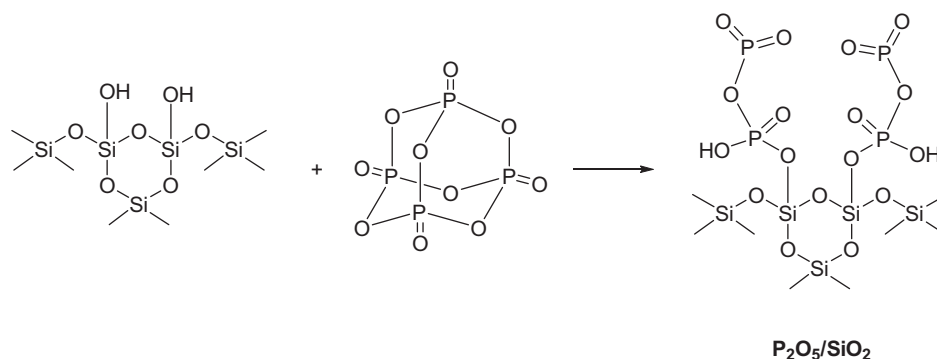


Table 1. Optimization of reaction conditions for synthesis of compound 4c catalyzed by P₂O₅/SiO₂^a

Entry	Catalyst (g)	Solvent	T (°C)	Time (min)	Isolated yield (%)
1	-	-	90	90	Trace
2	-	H ₂ O	Reflux	90	Trace
3	-	EtOH	Reflux	90	Trace
4	0.01	-	70	8	48
5	0.01	-	90	7	59
6	0.01	-	110	8	59
7	0.02	-	70	8	68
8	0.02	-	90	7	73
9	0.02	-	110	6	74
10	0.04	-	70	8	80
11	0.04	-	90	5	84
12	0.04	-	110	6	83
13	0.05	-	70	6	88
14	0.05	-	90	2	94
15	0.05	-	110	4	93
16	0.07	-	90	4	92
17	0.07	-	110	2	92
18	0.05	H ₂ O	Reflux	20	16
19	0.05	MeOH	Reflux	20	22
20	0.05	EtOH	Reflux	20	30
21	0.05	CH ₃ CN	Reflux	20	15
22	0.05	CH ₂ Cl ₂	Reflux	20	20

^aReaction conditions: β-naphthol (1) (1 mmol), 4-chlorobenzaldehyde (2c) (1 mmol), and methyl carbamate (3) (1.1 mmol).

Table 2. Synthesis of carbamatoalkyl naphthols (4a-i) using P₂O₅/SiO₂^a

Entry	R	Product	Time (min)	Isolated yield (%)	m.p. (°C)	
					Found	Reported
1	2-ClC ₆ H ₄	4a	4	91	180–182	181–183 (Ghashang, 2014)
2	3-ClC ₆ H ₄	4b	2	93	202–204	200–202 (Ghashang, 2014)
3	4-ClC ₆ H ₄	4c	2	94	202–203	203–205 (Ghashang, 2014)
4	2,4-Cl ₂ C ₆ H ₃	4d	2	92	191–193	189–191 (Ghashang, 2014)
5	4-BrC ₆ H ₄	4e	3	89	196–199	197–199 (Ghashang, 2014)
6	4-FC ₆ H ₄	4f	4	90	204–205	203–205 (Ghashang, 2014)
7	3-O ₂ NC ₆ H ₄	4g	3	94	254–256	249–251 (Zare et al., 2012)
8	4-O ₂ NC ₆ H ₄	4h	3	95	200–202	203–205 (Zare et al., 2012)
9	4-MeC ₆ H ₄	4i	4	88	186–188	187–189 (Ghashang, 2014)

^aReaction conditions: β-naphthol (1) (1 mmol), an aromatic aldehyde (2a-i) (1 mmol), methyl carbamate (3) (1.1 mmol), P₂O₅/SiO₂ (0.05 g), 90°C, solvent-free.

(94, 93, 92, 92, and 91% yields for first to fifth use, respectively) which clearly demonstrates the practical reusability of this catalyst. This reusability demonstrates the high stability and turnover of P₂O₅/SiO₂ under the employed conditions. The stability of P₂O₅/SiO₂ has been also confirmed in several papers reviewed by Eshghi and Hassankhani (2012) and also two papers reported by Habibi et al. (2013) and Eshghi, Rahimizadeh, Ghadamyari, and Shiri (2012). While P₂O₅ is very sensitive to moisture, P₂O₅/SiO₂ is stable in various reaction mixtures containing water, amines and alcohols.

3. Conclusion

In this paper, a simple, efficient, and eco-friendly method for the synthesis of carbamatoalkyl naphthols by the one-pot three-component reaction of β -naphthol with a wide range of aromatic aldehydes and methyl carbamate using P_2O_5/SiO_2 as catalyst has been successfully developed. The method was fast and the desired products were obtained within a few minutes in high yields under solvent-free conditions at 90°C. The catalyst can be recycled after a simple work-up, and used at least five times without substantial reduction in its catalytic activity. The procedure is also advantageous in the sense that it is a solvent-free reaction and therefore operates under environmentally friendly conditions.

4. Experimental

All chemicals were available commercially and used without additional purification. Melting points were recorded on a Stuart SMP3 melting point apparatus. The IR spectra were obtained using a Tensor 27 Bruker spectrophotometer as KBr disks. The 1H NMR spectra were recorded with a Bruker 300 FT spectrometer.

4.1. Preparation of P_2O_5/SiO_2

A mixture of P_2O_5 (3 g) and SiO_2 (4 g, 230–400 mesh) was ground vigorously in a mortar for 30 min to give P_2O_5/SiO_2 as a white powder (Eshghi et al., 2001).

4.2. General procedure for the synthesis of carbamatoalkyl naphthols (4a-i) catalyzed by P_2O_5/SiO_2

A mixture of β -naphthol (**1**) (1 mmol), an aromatic aldehyde (**2a-i**) (1 mmol), methyl carbamate (**3**) (1.1 mmol), and P_2O_5/SiO_2 (0.05 g) was heated in the oil bath at 90°C for 2–4 min and monitored by TLC. On completion of the transformation, the reaction mixture was cooled to room temperature and hot ethanol was added. The catalyst was collected by filtration, and the filtrate was cooled to room temperature. The crude product was collected and recrystallized from ethanol to give compounds **4a-i** in high yields.

4.3. Selected spectral data

Methyl (3-chlorophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate (**4b**): IR (KBr disc): ν 3417 (NH), 3293 (OH), 1690 (C=O) cm^{-1} ; 1H NMR (300 MHz, DMSO- d_6): δ 3.59 (s, 3H, OCH₃), 6.87 (d, 1H, J = 5.7 Hz, CH), 7.16 (d, 1H, J = 6.9 Hz, arom-H), 7.20–7.33 (m, 6H, arom-H and NH), 7.42 (t, 1H, J = 7.2 Hz, arom-H), 7.81 (t, 2H, J = 8.4 Hz, arom-H), 7.92 (d, 1H, J = 8.4 Hz, arom-H), 10.24 (br, 1H, OH).

Methyl (4-chlorophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate (**4c**): IR (KBr disc): ν 3421 (NH), 3212 (OH), 1686 (C=O) cm^{-1} ; 1H NMR (300 MHz, DMSO- d_6): δ 3.58 (s, 3H, OCH₃), 6.83 (broadened doublet, 1H, CH), 7.18–7.36 (m, 6H, arom-H), 7.41 (t, 1H, J = 7.0 Hz, arom-H), 7.71 (br, 1H, NH), 7.76–7.85 (m, 2H, arom-H), 7.90 (d, 1H, J = 9.0 Hz, arom-H), 10.20 (br, 1H, OH).

Methyl (2,4-dichlorophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate (**4d**): IR (KBr disc): ν 3402 (NH), 3259 (OH), 1678 (C=O) cm^{-1} ; 1H NMR (300 MHz, DMSO- d_6): δ 3.55 (s, 3H, OCH₃), 6.82 (broadened doublet, 1H, CH), 7.14 (d, 1H, J = 9.0 Hz, arom-H), 7.29 (t, 1H, J = 7.2 Hz, arom-H), 7.37–7.57 (m, 4H, arom-H), 7.77 (d, 1H, J = 8.7 Hz, arom-H), 7.82 (d, 1H, J = 7.5 Hz, arom-H), 7.95 (br, 1H, NH), 8.01 (d, 1H, J = 8.7 Hz, arom-H), 9.99 (br, 1H, OH).

Methyl (4-bromophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate (**4e**): IR (KBr disc): ν 3417 (NH), 3288 (OH), 1688 (C=O) cm^{-1} ; 1H NMR (300 MHz, DMSO- d_6): δ 3.58 (s, 3H, OCH₃), 6.82 (broadened doublet, 1H, CH), 7.18 (d, 2H, J = 8.4 Hz, arom-H), 7.23 (d, 1H, J = 9.0 Hz, arom-H), 7.45 (t, 1H, J = 8.4 Hz, arom-H), 7.37–7.50 (m, 3H, arom-H), 7.70–7.85 (m, 3H, arom-H and NH), 7.90 (d, 1H, J = 7.8 Hz, arom-H), 10.12 (br, 1H, OH).

Methyl (4-fluorophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate (**4f**): IR (KBr disc): ν 3422 (NH), 3224 (OH), 1685 (C=O) cm^{-1} ; 1H NMR (300 MHz, DMSO- d_6): δ 3.58 (s, 3H, OCH₃), 6.85 (d, 1H, J = 9.0

Hz, CH), 7.09 (t, 2H, $J = 8.1$ Hz, arom-H), 7.20–7.45 (m, 5H, arom-H), 7.70–7.85 (m, 3H, arom-H and NH), 7.92 (d, 1H, $J = 8.1$ Hz, arom-H), 10.23 (br, 1H, OH).

Methyl (2-hydroxynaphthalen-1-yl)(4-nitrophenyl)methylcarbamate (**4h**): IR (KBr disc): ν 3422 (NH), 3268 (OH), 1683 (C=O), 1517 and 1345 (NO_2) cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 3.61 (s, 3H, OCH_3), 6.96 (d, 1H, $J = 7.5$ Hz, CH), 7.23 (d, 1H, $J = 9.0$ Hz, arom-H), 7.30 (t, 1H, $J = 7.5$ Hz, arom-H), 7.36–7.52 (m, 3H, arom-H), 7.80–7.95 (m, 4H, arom-H and NH), 8.16 (d, 2H, $J = 8.7$ Hz, arom-H), 10.26 (br, 1H, OH).

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Supplemental data

Supplemental data for this article can be accessed at <http://dx.doi.org/10.1080/23312009.2017.1317582>.

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