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Solid lithium perchlorate-mediated conjugate addition of thiols and indoles to α,β-unsaturated carbonyl compounds

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RESEARCH ARTICLE

Solid lithium perchlorate-mediated conjugate addition of thiols and indoles to α,β-unsaturated carbonyl compounds

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A simple, efficient, and general synthetic strategy has been developed for the synthesis of Michael adducts in the presence of solid lithium perchlorate under solvent-free conditions. Nucleophilic addition of thiols and indoles gave the corresponding Michael adducts in high yields.

Keywords: Michael addition; Thiols; Indole; α,β-unsaturated olefins; Lithium perchlorate

1. Introduction

Adducts of thiols and indoles with electron-deficient olefins are important for their use as intermediates for the synthesis of pharmaceuticals and naturally occurring compounds [1–5]. For example, affinisine has been shown to produce delayed intention tremors, marked CNS depressant activity, ataxia, and hypothermia through its action at serotonin receptors, and a synthetic pentacyclic indole has used for its antitumor properties [6, 7]. Common approaches to the regioselective synthesis of 3-alkylated indoles involve the conjugate addition of indoles to α,β-unsaturated compounds in the presence of an acidic medium [8–10].

Conjugate additions promoted by Lewis acids have gained considerable attention due to their acceleration of carbon–carbon bond formation [11, 12]. Thus, numerous metal-based Lewis acid catalysts have been developed for Michael reactions, such as $AlCl_3$, $TiCl_4$, $SnCl_4$, $La(OTf)_3$, $InCl_3$, $FeCl_3$, $CeCl_3.7H_2O$–NaI [13, 14], $Sc(OTf)_3$ [15–17], $InBr_3$ [18, 19], $Bi(OTf)_3$ [20], $CuClO_4$ [21], $Cu(OTf)_2$ [22], K-10 [23], $Yb(OTf)_3$ [24], CdI_2 [25], $(DHQD)_2PYR$ [26], I_2 [27], n-Bu_4NBr [28], $NaAuCl_4{\cdot}2H_2O$ [29], and $[Al(DS)_3].3H_2O$ [30]. Unfortunately, many of the procedures employing these Lewis acids often require a large excess of reagents, long reaction times, and drastic reaction conditions in toxic solvents such as acetonitrile. Also, many of these Lewis acids are readily hydrolysed and cannot be recovered. Hence, we had a need to develop a more efficient, clean, and green process with an active catalyst.

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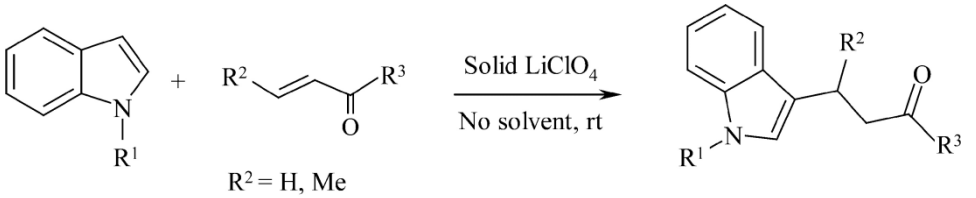
2. Results and discussion

Recently, $LiClO_4$ has emerged as a powerful promoter for various chemical processes, due to the remarkable tolerance of $LiClO_4$ toward strongly coordinating functional groups [31–36]. Herein, we describe the remarkable catalytic activity of $LiClO_4$ in the conjugate addition of indoles to α,β-unsaturated olefins using a catalytic amount of $LiClO_4$ (20 mol%) under solvent-free conditions and at room temperature. $LiClO_4$ is an inexpensive and readily available and recoverable catalyst in comparison to the other catalyst such as n-Bu_4NBr, $NaAuCl_4{\cdot}2H_2O$, $Yb(OTf)_3$, and $Sc(OTf)_3$. The mild reaction conditions, experimental simplicity, short reaction time, and high yields are the main advantages of using $LiClO_4$ in the present protocol.

The conjugate addition of indoles to α, β-unsaturated aldehydes and ketones in the presence of solid $LiClO_4$ gave the 3-oxoalkylation adduct in moderate to good yield and relatively short reaction time, without any dimerization or polymerization to side products, scheme 1. It is well known that reactions carried out under solvent-free conditions are desirable due to easy work-up and experimental conditions. Also, in these reactions the use of organic solvent is reduced [37, 38].



SCHEME 1

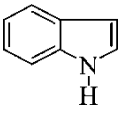
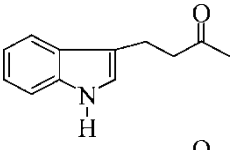
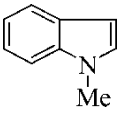
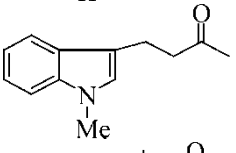
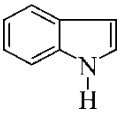
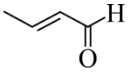
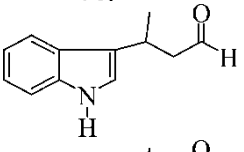
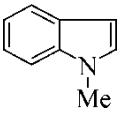
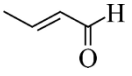
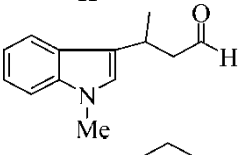
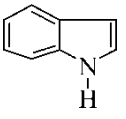
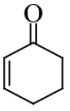
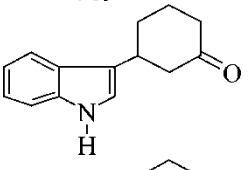
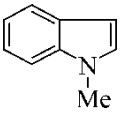
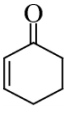
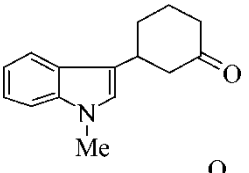
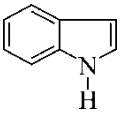
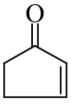
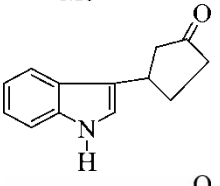
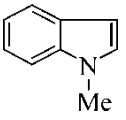
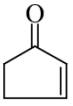
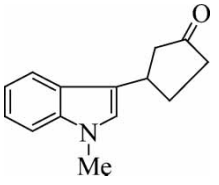
The data in table 1 show that the reaction of indole and *N*-methylindole with Michael acceptors affords the corresponding Michael adducts in moderate to good yields. In all cases the reactions smoothly proceed at room temperature with high regioselectivity. No 1,2-adduct or *N*-alkylated indoles were obtained under these reaction conditions. We have considered that the electronic properties of the aromatic ring have an effect on the rate of the reaction. In some cases the rate of the reaction was accelerated for *N*-methylated indoles, which is in full expectation with the increased nucleophilicity of an indole bearing an electron-donating group. For example, the reaction time of *N*-methylindole with methyl vinyl ketone was shorter than that for indole, table 1, entries 1 and 2.

A longer reaction time does not increase the yield or the extent of the reaction; however, the reaction mixture consists only of the starting materials and the product, which are separable by column chromatography. This chemistry was also carried out by 10 mol% of solid $LiClO_4$ under solvent-free conditions and at room temperature, but a longer reaction time is needed to achieve the same yield as in the case of using 20 mol%.

The conjugate addition of thiols to α,β-unsaturated carbonyl compounds has been carried out in the presence of acidic and basic media [18, 19], but there is a great need for more efficient and environmentally friendly catalysts. In the course of our research on the application of $LiClO_4$ in organic synthesis, we found that $LiClO_4$ was an effective accelerator in the conjugated addition of thiols to α,β-unsaturated olefins under solvent-free conditions.

To show the generality of this method, a wide range of α,β-unsaturated olefins were subjected to this procedure and converted into the corresponding Michael adduct with high yields,

Table 1. Michael addition of indoles to a,b-unsaturated compounds under solvent-free conditions.

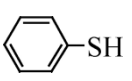
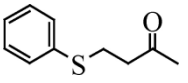
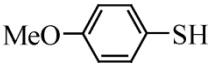
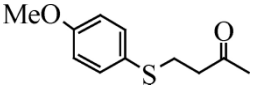
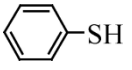
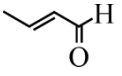
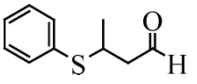
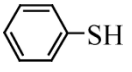
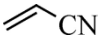
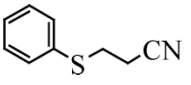
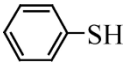
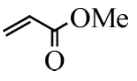
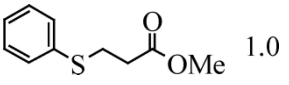
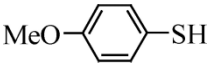
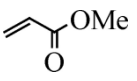
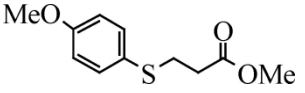
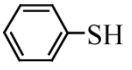
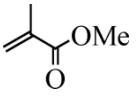
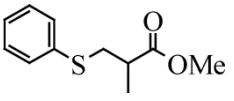
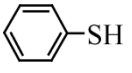
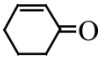
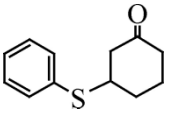
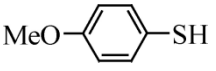
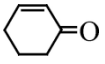
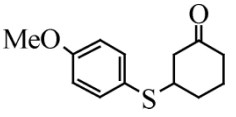
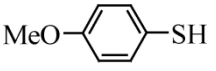
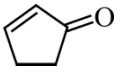
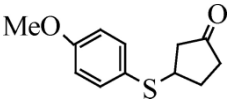
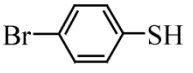
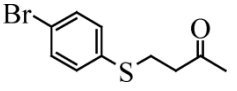
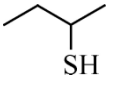
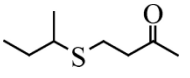
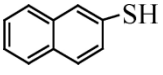
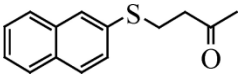
Entry	Substrate	Enone	Product	Time (h)	Yield(%)[a]
1				1.5	90
2				1.0	94
3				3.0	55
4				3.0	55
5				5.0	51
6				5.5	54
7				5.5	52
8				6	50

[a] Isolated yield.

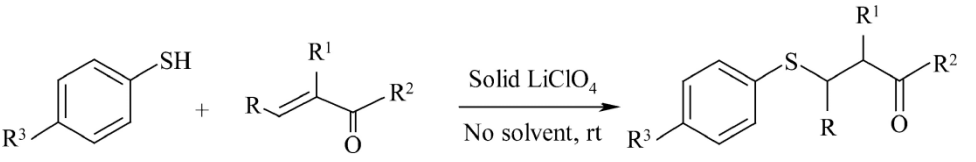
with some of the examples generalized in scheme 2. On the whole thiols react faster than indoles, with higher conversion under the same reaction conditions. Table 2 summarizes the conjugate addition of various thiols to cyclic and acyclic enones under optimized conditions. Slight modification of the substrate influenced the yield of the reaction.

Although we have not yet studied the mechanism of the reaction in detail, preliminary experiments do provide meaningful information about the value of the catalyst. The role of $LiClO_4$

Table 2. Michael addition of thiophenol to α,β-unsaturated compounds under solvent-free conditions.

Entry	Substrate	Enone	Product	Time (h)	Yield(%)[a]
1				1.0	95
2				0.5	97
3				1.5	71
4				1.0	78
5				1.0	95
6				1.0	96
7				1.5	82
8				2.0	65
9				2.0	72
10				2.0	75
11				1.2	94
12				1.0	96
13				1.0	95

[a]Isolated yield.



R^3 = H, OMe, Br

R = H, Me
R^1 = H, Me
R^1, R^2 = -$(CH_2)_3$-, -$(CH_2)_2$-
R^2 = H, Me, OMe

SCHEME 2

in mediating conjugate addition maybe realized through the coordination of the carbonyl group to Li^+, so activating the carbon–carbon double bond under solvent-free conditions.

In conclusion, we have developed a simple and efficient procedure for promoting conjugate addition of indoles and thiols to α,β-unsaturated olefins under solvent-free conditions. The availability of inexpensive starting materials along with simplicity and green methodology provide an efficient method for the synthesis of Michael adducts in short time and with good yields.

3. Experimental

All reactions were carried out under an atmosphere of argon. NMR spectra were recorded on a Bruker ACF 500 using chloroform-d as solvent. IR spectra were measured on a Perkin-Elmer 1600 FT-IR spectrometer. Column chromatography was performed on silica gel, Merck grade 60. CH_2Cl_2 or diethyl ether was distilled before use. Lithium perchlorate was purchased from Acros. All other chemicals were purchased from Fluka and Merck. Light petroleum refers to the fraction with distillation range 60–80 °C.

Caution: Although we did not have any accident while using $LiClO_4$, it is advisable to dry lithium perchlorate in a fume hood using a suitable lab-shield.

3.1 *General procedure for the Michael addition of indoles and thiols to α,β-unsaturated olefins*

To a mixture of $LiClO_4$ (1 mmol) and methyl vinyl ketone (5 mmol) was added an indole or thiol (5 mmol). The reaction mixture was stirred at room temperature for the appropriate time (tables 1 and 2). After completion of the reaction, CH_2Cl_2 (20 mL) was added, and the $LiClO_4$ was removed by filtration. After washing with water (2 × 20 mL), the solvent was evaporated off and the product was isolated in almost pure form in some cases. Further purification was carried out by column chromatography on silica gel, eluting with ethyl acetate/light petroleum. All compounds were characterized by their IR, NMR, and MS spectra.

3.2 *4-(3-Indolyl)-2-butanone* [24]

Yield 90%; white solid, mp 93–95 °C; ^{1}H NMR ($CDCl_3$) δ2.18 (s, 3H), 2.98 (t, J = 7.5 Hz, 2H), 3.10 (t, J = 7.5 Hz, 2H), 7.01 (m, 1H), 7.11–8.00 (m, 5H). IR (KBr) υ 3315, 2908, 1702, 1474, 1461, 736 cm^{-1}.

3.3 *4-(3-N-Methylindolyl)-2-butanone* [24]

Yield 94%; oil; ^{1}H NMR ($CDCl_3$) δ 2.19 (s, 3H), 2.87 (t, $J = 7.4$ Hz, 2H), 3.09 (t, $J = 7.4$ Hz, 2H), 3.79 (s, 3H), 6.88 (s, 1H), 7.15 (td, $J = 7.3$, 0.4 Hz, 1H), 7.26 (td, $J = 7.4$, 0.4 Hz, 1H), 7.31 (dd, $J = 8.6$, 0.4 Hz, 1H), 7.61 (dd, $J = 8.1$, 0.4 Hz, 1H).

3.4 *3-(3-Indolyl)butanal*

See table 1, entry 3 [9].

3.5 *3-(3-N-Methylindolyl)butanal*

See table 1, entry 4 [9].

3.6 *3-(3-Indolyl)cyclohexanone*

See table 1, entry 5 [10, 24].

3.7 *3-(3-N-Methylindolyl)cyclohexanone*

See table 1, entry 6 [10, 24].

3.8 *3-(3-Indolyl)cyclopentanone*

See table 1, entry 7 [13, 24, 43].

3.9 *3-(3-N-Methylindolyl)cyclopentanone*

See table 1, entry 8 [24].

3.10 *4-Phenylthio-2-butanone* [28]

Yield 95%; oil; ^{1}H NMR ($CDCl_3$) δ 2.18 (s, 3H), 2.77 (t, $J = 7.3$ Hz, 2H), 3.17 (t, $J = 7.3$ Hz, 2H), 7.18–7.38 (m, 5H).

3.11 *4-(4-Methoxyphenylthio)-2-butanone*

Yield 97%; yellow oil; ^{1}H NMR ($CDCl_3$) δ 2.17 (s, 3H), 2.59 (t, $J = 7.4$ Hz, 2H), 2.91 (t, $J = 7.4$ Hz, 2H), 3.67 (s, 3H), 6.76 (dm, $J = 10.2$ Hz, 2H), 7.21 (dm, $J = 10.2$ Hz, 2H). ^{13}C NMR ($CDCl_3$) δ 29.5, 29.6, 42.8, 54.9, 114.2, 114.3, 125.1, 128.7, 133.1, 133.4, 206.4. MS 210 (M^+), 153, 140, 125, 108, 96, 71, 43 (100). IR (KBr) υ 3056, 2943, 1716, 1593, 1247, 1183, 1033, 746 cm^{-1}.

3.12 *3-Phenylthiobutanal* [28, 40]

Oil; ^{1}H NMR ($CDCl_3$) δ 1.36 (d, $J = 6.8$ Hz, 3H), 2.54–2.60 (ddd, $J = 17.3$, 9.5, 1.5 Hz, 1H), 2.66–2.71 (ddd, $J = 17.2$, 9.5, 1.5 Hz, 1H), 3.69 (m, 1H), 7.25–7.33 (m, 3H), 7.42–7.43 (m, 2H), 9.73 (t, $J = 1.5$ Hz, 1H).

3.13 *Cyano-2-phenylthioethane*

See table 2, entry 4 [28, 40, 41].

3.14 *Methyl 3-(phenylthio)propionate* **[28, 39]**

Colorless oil; ^{1}H NMR ($CDCl_3$) δ 2.45 (t, $J = 7.4$ Hz, 2H), 3.03 (t, $J = 7.4$ Hz, 2H), 7.03–7.06 (m, 1H), 7.11–7.14 (m, 2H), 7.27–8.00 (m, 2H). IR (KBr) υ 3031, 2951, 1739, 1581, 1479, 1441, 1404, 1250, 1174, 1021, 772 cm^{-1}.

3.15 *Methyl 3-(4-methoxyphenylthio)propionate*

Yellow oil; ^{1}H NMR ($CDCl_3$) δ 2.48 (t, $J = 7.3$ Hz, 2H), 2.96 (t, $J = 7.2$ Hz, 2H), 3.58 (s, 3H), 3.71 (s, 3H), 6.76 (dm, $J = 8.8$ Hz, 2H), 7.28 (dm, $J = 8.8$ Hz, 2H). ^{13}C NMR ($CDCl_3$) δ 30.9, 34.2, 51.5, 55.1, 114.5, 114.8, 124.9, 126.3, 128.1, 134.0, 174.5. MS 226 (M^+, 100), 195, 166, 153, 139, 124, 108, 96, 87, 69, 59, 45. IR (KBr) υ 2954, 1737, 1599, 1497, 1439, 1247, 916, 729 cm^{-1}.

3.16 *Methyl 2-methyl-3-(phenylthio)propionate*

See table 2, entry 7 [28, 39].

3.17 *3-Phenylthiocyclohexanone*

See table 2, entry 8 [28, 26].

3.18 *3-(4-Methoxylphenylthio)cyclohexanone* **[42]**

Colorless oil; ^{1}H NMR ($CDCl_3$) δ 1.57–1.58 (m 2H), 2.01–2.02 (m, 2H), 2.22–2.23 (m, 3H), 2.49–2.54 (m, 1H), 3.14 (m, 1H), 6.77 (dm, $J = 8.3$ Hz, 2H), 7.28 (dm, $J = 8.4$ Hz, 2H). ^{13}C NMR ($CDCl_3$) δ 23.7, 30.9, 40.5, 46.7, 47.4, 55.0, 114.3, 114.5, 122.6, 132.3, 136.1, 159.7, 208.7. MS 236 (M^+), 140 (100), 125, 97, 69, 55, 41. IR (KBr) υ 2954, 1716, 1591, 1492, 1252, 1173, 734 cm^{-1}.

3.19 *3-(4-Methoxyphenylthio)cyclopentanone* **[42]**

Colorless oil; ^{1}H NMR ($CDCl_3$) δ 2.11–2.45 (m, 6H), 3.65 (m, 1H), 3.68 (s, 3H), 6.73 (dm, $J = 8.1$ Hz, 2H), 7.27 (dm, $J = 8.2$ Hz, 2H). ^{13}C NMR ($CDCl_3$) δ 28.8, 36.4, 44.3, 44.7, 55.0, 114.2, 114.3, 123.6, 132.9, 134.9, 135.5, 216.2. MS 222 (M^+), 140 (100), 125, 96, 83, 69, 55, 39. IR (KBr) υ 3045, 2965, 1743, 1593, 1492, 1247, 734 cm^{-1}.

3.20 *4-(4-Bromothiophenyl)-2-butanone*

Yield 94%; Yellow oil; ^{1}H NMR ($CDCl_3$) δ 1.95 (s, 3H), 2.55 (t, $J = 7.2$ Hz, 2H), 2.90 (t, $J = 7.4$ Hz, 2H), 7.01 (dm, $J = 8.6$ Hz, 2H), 7.21 (dm, $J = 8.6$ Hz, 2H). IR (KBr) υ 3476, 2936, 1715, 1476, 1369, 1246, 1100, 800 cm^{-1}.

3.21 *4-(Butan-2-ylthio)-2-butanone*

Yield 96%; **w**hite oil; ^{1}H NMR ($CDCl_3$) δ 0.72 (t, $J = 7.3$ Hz, 3H), 1.00 (d, $J = 6.8$ Hz, 3H), 1.23–1.37 (m, 2H), 1.91 (s, 3H), 2.41–2.50 (m, 5H). ^{13}C NMR ($CDCl_3$) δ 11.6, 21.0, 24.2, 29.7, 29.8, 42.4, 44.1, 206.1. IR (KBr) υ 2961, 1717, 1463, 1369, 1230, 1161, 923, 800, 738 cm^{-1}.

3.22 *4-(2-Naphthylthio)-2-butanone*

Yield 95%; light brown solid; mp 57.3–58.6 °C; ^{1}H NMR ($CDCl_3$) δ 2.04 (s, 3H), 2.71 (t, $J = 7.2$ Hz, 2H), 3.18 (t, $J = 7.2$ Hz, 2H), 7.36–7.43 (m, 3H), 7.69–7.74 (m, 4H). ^{13}C NMR ($CDCl_3$) δ 26.5, 30.5, 43.2, 126.2, 127.1, 127.4, 128.2, 128.9, 132.2, 133.9, 134.2, 206.9. IR (KBr) υ 3423, 3046, 2923, 1715, 1623, 1415, 1269, 1064, 916, 746 cm^{-1}.

Acknowledgement

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