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Short Communication

Amberlyst-15©: An efficient heterogeneous reusable catalyst for selective anti-Markovnikov addition of thiols to alkenes/alkynes and for thiolysis of epoxides

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ABSTRACT

The anti-Markovnikov addition of thiols to alkenes/alkynes and thiolysis of epoxides is described using Amberlyst-15© as a selective, commercially available, inexpensive and reusable catalyst. The products like diorganyl sulphides, β-hydroxy sulphides and phenyl(styryl)sulfanes were obtained in good to excellent yields in short reaction time and with high regio-selectivity. The catalyst was reused up to five consecutive recycles without any loss in its catalytic activity. The developed methodology is a metal free protocol for C–S bond formation reaction with high atom economy.

1. Introduction

Thioethers, thioesters, β-hydroxy sulphides and phenyl(styryl) sulfanes are important building blocks in several natural products, pharmaceutically active compounds and various intermediates [1–4]. The β-hydroxy sulphides are useful in the synthesis of benzoxathiepines [5,6], benzodiazepines, [7,8] α-substituted α, β-unsaturated enones [9,10], α-thioketones, [11] β-hydroxysulfoxides [12–14] and allylic alcohols [15]. The β-hydroxy sulphides are also important compounds in various intermediates and generally prepared via thiolysis of 1,2-epoxides [16]. The synthesis of thioethers via nucleophilic addition of thiols to electron deficient alkenes is catalyzed by Lewis acid as well as protics [17–20]. The addition of thiols or thiolate anions to carbon–carbon double bonds has a major drawback of polymerization of thiols under acidic conditions [18]. Conventionally, these compounds are synthesized by the addition of thiols, thiolate anions to organic halides, alkenes and alkynes in the presence of different reagents/catalysts. Markovnikov addition to styrenes is a common reaction [21], whereas H-rho-zeolite and benzene-reflux [22,23], silica nano particles [24], $CeCl_3$ [25], InI [26] and acidic ionic liquids [27] are known to lead anti-Markovnikov products. The synthesis of thioethers and β-hydroxysulfides is usually carried out by using thiols under basic or acidic conditions or in the presence of promoters and/or catalysts.

The utility of conventional protocols which comprises the use of non-greener homogeneous acid catalysis is limited as it suffers from one or more drawbacks like lower yield, longer reaction time, use of expensive metal catalyst, lower selectivity and without consideration of recyclability aspect. Hence, developing heterogeneous catalysis is an attractive alternative to these conventional catalysis [28–33]. Furthermore, they can be recycled over a prolonged period without any difficulty in handling and storage.

In this context, to develop an environmentally benign and reusable protocol for the synthesis of thioethers, β-hydroxysulphides and one pot synthesis of phenyl(styryl)sulfanes is obligatory. Herein, we report catalytic efficiency of Amberlyst-15© as a metal-free protocol for the anti-Markovnikov addition of thiol towards styrene and thiolysis of 1,2 epoxides (Scheme 1).

2. Experimental

2.1. Materials

All chemicals and reagents were procured from Sigma Aldrich, S.D. Fine chemical, Lancaster (Alfa-Aesar), all other reagents and solvents were of analytical grade and were used without further purification.

2.2. Characterization methods

The products were characterized using ^{1}H NMR spectra (Varian Mercury 300 NMR Spectrometer) and IR (Perkin-Elmer FT-IR)

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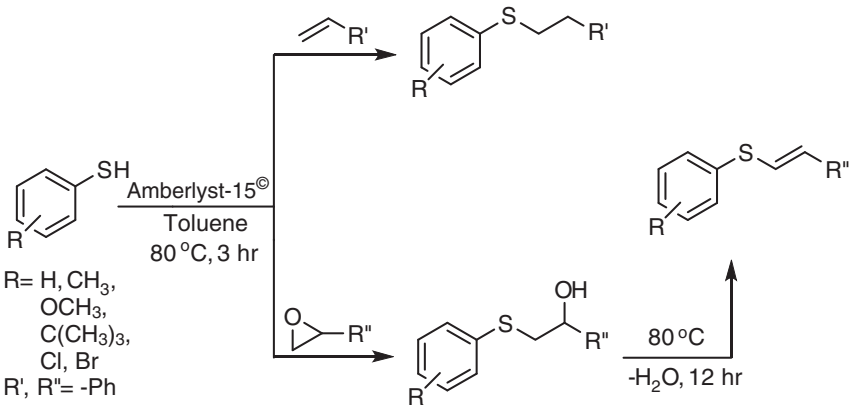


Scheme 1. Amberlyst-15© catalyzed anti-Markovnikov addition of thiols to alkenes and thiolysis of epoxides.

spectroscopic techniques. The reaction progress was monitored using GC analysis (Perkin-Elmer, Clarus 400) (BP-10 GC column, 30 m × 0.32 mm ID, film thickness 0.25 mm) equipped with flame ionization detector (FID) or thin layer chromatography using the Merck silica gel 60 F254 plates. The yields reported in Tables 1, 2 and 4 are GC yields and were calculated using external standard method, Tables 3 and 5 are isolated yield. The products were confirmed by GC-MS (Shimadzu GC-MS QP 2010 Rtx-17, 30 m × 25 mm ID, film thickness 0.25 μm df) (column flow 2 mLmin^{-1}, 80 °C to 240 °C at 10 °C/min rise). Some representative products were also confirmed by ^{1}H and ^{13}C NMR. All products are known in the literature.

2.3. General experimental procedure for anti-Markovnikov addition of thiol with alkene and thiolysis of epoxides

Toluene (2 ml), alkene (0.5 mmol) and thiol (2.0 mmol)/epoxide(0.5 mmol) and thiol (1.2 mmol), and Amberlyst-15© (30 wt%) were added to a 10 ml sealed tube. The reaction mixture was heated to 80 °C for a desired time and after completion of reaction, it was cooled to room temperature. 10% NaOH (5 ml) was added to the reaction mixture. The catalyst (Amberlyst-15©) was separated by normal filtration technique and product was extracted with ethyl acetate (3 × 5 ml). Alkene/epoxide conversion and product formation were monitored using gas chromatography. The residue obtained was purified by column chromatography (silica gel, 60–120 mesh; PE–EtOAc, 95:05) to afford desired thioethers, β-hydroxy sulphides and phenyl(styryl)sulfanes products. The structures of the products were confirmed by GC-MS, ^{1}H NMR, and IR spectroscopic techniques. The purity of compounds was determined by GC-MS analysis (see supporting information).

3. Results and discussion

3.1. Anti-Markovnikov addition of thiol with alkene

Initially, anti-Markovnikov addition of thiols to styrene was selected as a model reaction and the influence of reaction parameter such as catalyst screening, solvent effect, catalyst loading, the mole ratio of substrates, reaction temperature and time was studied (Table 1). Various acid catalysts such as *p*-TSA, Amberlyst-15©, Montmorillonite K-10 and neat were screened (Table 1, entries 1–4). We observed that Amberlyst-15© provides 78% yield of the desired product (Table 1, entry 2) under screening conditions. The reaction was also carried out in the absence of a catalyst; however no product formation was observed. This confirms the necessity of Amberlyst-15© to catalyze the reaction with higher yields (Table 1, entry 4).

Considering the activity of Amberlyst-15©, we checked the effect of different types of commercially available ion exchange resins like gel type (Amberlite-IR120), macroreticular porous having polystyrene backbone like Indion-130 and non-porous perfluorinated resin sulfonic acid (Nafion-NR50) were examined for the entitled reaction (Table 2, entries 1–4). It can be seen that Amberlyst-15© was the most active catalyst for anti-Markovnikov addition of thiols to styrene as compared to other ion exchange resins. It is also observed that Nafion-NR50, inspite

Table 1
Optimization of reaction parameters for anti-Markovnikov addition of thiol with styrene.[a]

Entry	Catalyst	Solvent	Temp (°C)	Time (h)	Conv. (%)[b]	Yield (%)[b]
Catalyst screening						
1	*p*-TSA	neat	80	6	50	48
2	Amberlyst-15©	neat	80	6	100	78
3	Mont K-10	neat	80	6	100	70
4	none	neat	80	6	n. r.	–
Solvent study						
5	Amberlyst-15©	CH3CN	80	6	n. r.	–
6	Amberlyst-15©	1, 4-dioxane	80	6	n. r.	–
7	Amberlyst-15©	toluene	80	6	100	90
Effect of catalyst loading						
8[c]	Amberlyst-15©	toluene	80	6	80	78
9[d]	Amberlyst-15©	toluene	80	6	100	90
10[e]	Amberlyst-15©	toluene	80	6	100	90
Effect of temperature						
11	Amberlyst-15©	toluene	60	6	90	85
12	Amberlyst-15©	toluene	80	6	100	90
13	Amberlyst-15©	toluene	100	6	100	90
14	Amberlyst-15©	toluene	RT	6	n. r.	–
Effect of mole ratio						
15[f]	Amberlyst-15©	toluene	80	6	100	75
16[g]	Amberlyst-15©	toluene	80	6	100	98
17[h]	Amberlyst-15©	toluene	80	6	100	58
18[i]	Amberlyst-15©	toluene	80	6	100	90
19[j]	Amberlyst-15©	toluene	80	6	100	15
Time study						
20	Amberlyst-15©	toluene	80	6	100	98
21	Amberlyst-15©	toluene	80	2	100	98
22	Amberlyst-15©	toluene	80	0.5	100	98
23	Amberlyst-15©	toluene	80	0.16	100	95

[a] Reaction conditions: styrene (0.5 mmol), thiol (1.5 mmol), catalyst (30 wt%), temperature (80 °C).
[b] GC yield.
[c] Catalyst (10 mg).
[d] Catalyst (15 mg).
[e] Catalyst (20 mg).
[f] Styrene:Thiol (1:2).
[g] Styrene:thiol (1:4).
[h] Styrene:Thiol (1:1).
[i] Styrene:Thiol (1:3).
[j] Styrene:Thiol (4:1).

Table 2
Effect of various ion exchange resins on anti-Markovnikov addition of thiol with styrene.[a]

Entry	Catalyst	H^+ capacity (meq/g)	Surface area (m^2/g) (Langmuir)	Particle size (micron)	Average pore dia (A°)	Yield[b] (%)
1	Amberlite-IR120	4.4	–	–	–	45
2	Amberlyst-15©	4.7	44	600–800	260	78
3	Nafion-NR50	0.8	<0.02	–	–	76
4	Indion-130	4.8	–	420–1200	–	54

[a] Reaction conditions: Styrene (0.5 mmol), Thiol (1.5 mmol), Catalyst (30 wt%), Temperature (80 °C).
[b] GC yield.

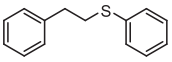
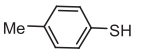
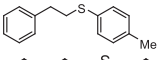
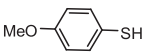
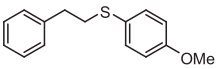
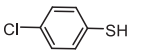
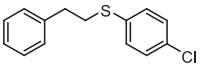
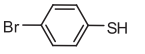
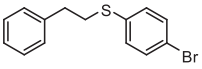
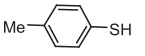
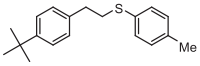
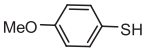
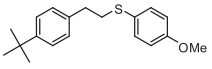
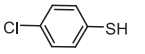
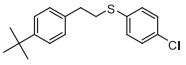
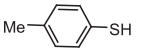
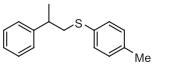
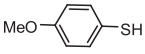
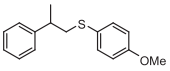
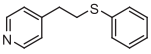
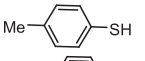
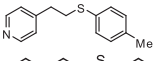
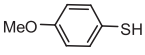
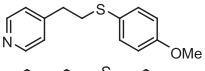
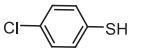
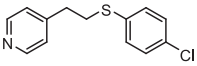
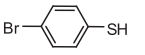
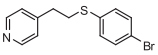
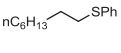
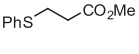
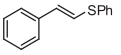
of its surface area and lower H^+ exchange capacity gives comparatively similar results as that of Amberlyst-15©. (Table 2, entries 2–3) Nafion-NR50 is a type of perfluorinated resin sulfonic acids with a very high acidity values, e.g. Hammet acidity function in the range of −11 to −13 (equivalent to 100% sulfuric acid) whereas Amberlyst-15© is a resin composed of styrene/divinylbenzene copolymer with sulfonic acid type functionality, and the probable reason for its higher activity can be explained on the basis of its physical properties, such as reasonably good surface area (Langmuir 44 m^2/g, and BET 29.52 m^2/g) and excellent H^+ capacity (4.7 meq/g based on Hammet scale) [29,30]. Considering the higher cost of Nafion-NR50, Amberlyst-15© was chosen and used for further studies.

Initially, the reaction was performed without any solvent and it gave 78% yield for the Amberlyst-15© catalyst. We also checked the effect of various solvents. We did not observe any conversion in acetonitrile as well as in 1, 4-dioxane (Table 1, entries 5–6). In case of toluene the reaction was more favourable and gave 90% yield of desired product (Table 1, entry 7). In an attempt to determine the optimum catalyst loadings, various catalyst loadings ranging from 20 wt% to 40 wt% were used (Table 1, entries 8–10). The optimum results were observed with 30 wt% of catalyst (90% yield) and further increase in the catalyst loading had no significant impact on the yield of anti-Markovnikov addition product (Table 1, entry 9). To check the role of reaction temperature, we performed the reactions at various temperatures and was observed that the reaction works smoothly at 80 °C with an appreciable yield (Table 1, entries 15–19). The substrate ratio of styrene:thiol (1:4) was found to be optimum for the higher yields of desired product (Table 1, entries 11–14). The reaction parameters such as reaction time was also studied and observed to work within 0.5 h (Table 1, entries 20–23). Hence, the optimum reaction conditions for anti-Markovnikov addition of thiols with styrene are: catalyst- Amberlyst-15© (30 wt %), solvent-toluene (2 ml), at 80 °C for 0.5 h.

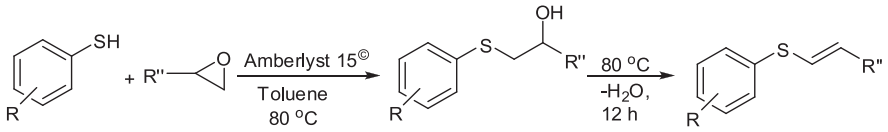
Several substituted thiophenols possessing various electron-withdrawing and electron-donating groups, including styrene derivatives were investigated for anti-Markovnikov addition reaction with styrene and were found to furnish good to excellent yields of the desired products (Table 3, entries 1–18) under optimum reaction conditions. We observed that the additions were regioselective and anti-Markovnikov in nature (Table 3, entries 1–5). Furthermore, the reaction of styrene derivatives such as 4-*tert*-butyl styrene and α-methyl styrene with different substituted thiophenols were also examined and was found to afford the respective thioethers in 79–91 % yield of desired products (Table 3, entries 6–10).

We have also studied the applicability of the developed catalytic system for the anti-Markovnikov addition of 4-vinyl pyridine with different substituted thiophenols including both electron-donating and electron-withdrawing group. We observed good to excellent yields of desired products under present reaction conditions (Table 3, entries 11–15). Alkene such as 1-octene was found to react with thiophenol to provide the octyl(phenyl)sulfane as a linear thioether in high yields (Table 3, entry 16). However, the addition of thiophenol with methyl acrylate goes into a Michael type addition mode to furnish the methyl 3-(phenylthio)propanoate as a addition product in excellent yield (Table 3, entry 17). Moreover, Amberlyst-15© effectively catalyzed the addition of thiophenol with phenyl acetylene was highly selective in

Table 3
Amberlyst-15© catalyzed anti-Markovnikov addition of thiophenol to alkene/alkyne.[a]

Entry	Thiols	Product	Time (h)	Yield[b](%)
Reactions of styrene with various thiols				
1			0.5	96
2			1	93
3			1	95
4			2	91
5			3	84
Reactions of 4-ter-butyl styrene with various thiols				
6			1	81
7			1	91
8			2	80
Reactions of α-methyl styrene with various thiols				
9			1.5	79
10			1.5	81
Reactions of 4-vinylpyridine with various thiols				
11			2	93
12			2	92
13			2	97
14			3	87
15			3	81
Reactions of oct-1-ene with thiophenol				
16			2	84
Reactions of methyl acrylate with thiophenol				
17			2	94
Reactions of phenyl acetylene with thiophenol				
18			2	93

[a] Reaction conditions: Alkene/Alkyne (0.5 mmol), Thiol (2.0 mmol), Amberlyst-15© (30 wt%), Toluene (2 mL), temperature (80 °C).
[b] Isolated yield.



Scheme 2. Amberlyst-15© catalyzed epoxides ring opening by thiols.

nature, which achieved 93% yield of (E)-phenyl(styryl)sulfane within 2 h (Table 3, entry 18).

3.2. Amberlyst-15© catalyzed epoxides ring opening by thiol

Encouraged with the successful application of the developed catalytic system for anti-Markovnikov addition of thiophenols with alkenes, we focused our attention for thiolysis of epoxides under the similar reaction conditions (Scheme 2) and we observed that the expected β-hydroxysulphides as a corresponding products with excellent yield. We also optimized the mole ratio of the styrene oxide:thiol and only 1.2 equivalent of thiol is necessary for the reaction (Table 4, entry 2), further increase in mole ratio does not affect the yield of β-hydroxysulphides (Table 4, entries 3–4). Considering the results obtained, we then explored the developed methodology for the thiolysis of various epoxides (Table 5).

We used present methodology for several substituted thiophenols possessing various electron-withdrawing and electron-donating groups, including various epoxides derivatives for thiolysis reaction and observed good to excellent yields of the desired products (Table 5, entries 1–10). Styrene oxide was treated under the optimized reaction conditions with various thiophenol derivatives, β-hydroxysulphides as a thiolysis product obtained regioselectivly and gave excellent yields (Table 5, entries 1–4) but it required longer reaction time as compared to anti-Markovnikov addition. Furthermore, we screened various electron donating as well as withdrawing thiophenols with 1,2-epoxycyclohexane gives a comparatively better yield within 4–5 hours (Table 5, entries 5–8). We have also extended the protocol for epichlorohydrin and gives very good results β-hydroxysulphides (Table 5, entries 9–10).

Furthermore, we run the reactions of various thiols with styrene oxide and epoxycyclohexane for longer reaction time and got a selective dehydration of β-hydroxysulphides to phenyl (styryl) sulfane with an excellent yield (Table 5, entries 11–16). This indicates that the methodology is applicable for the one pot synthesis of phenyl(styryl)sulfane.

3.3. Recyclability of catalyst

To make the protocol more economical and greener at established optimized reaction conditions, catalyst can be separated easily from the reaction mixture by simple filtration, wash with methanol and water and recycled further. It was observed that the catalyst can be efficiently recycled for five consecutive cycles with the same activity. There is no significant decrease in yield during the four recycles was observed. The yield declined up to 92% for the fifth cycle (Fig. 1).

Table 4
Effect of mole ratio on Amberlyst-15© catalysed epoxides ring opening by thiol.[a]

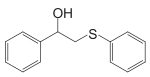
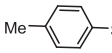
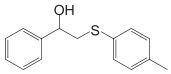
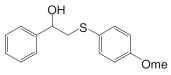
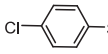
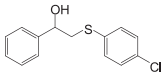
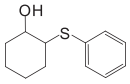
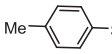
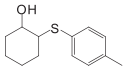
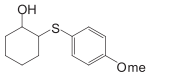
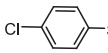
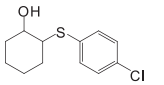
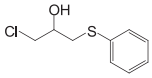
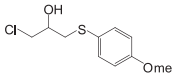
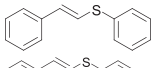
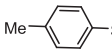
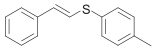
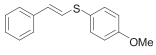
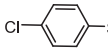
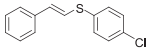
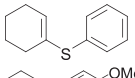
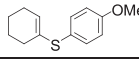
Entry	Styrene oxide: Thiol (mmol)	Solvent	Temp (°C)	Time (h)	Conv. (%)[b]	Yield (%)[b]
1	1:1	neat	80	6	91	88
2	1:1.2	neat	80	6	100	95
3	1:2	neat	80	6	100	96
4	1:4	neat	80	6	100	96

[a] Reaction conditions: catalyst (Amberlyst-15© -30 wt%), temperature (80 °C).
[b] GC yield.

4. Conclusion

In summary, we have developed a clean, safe and highly efficient metal free protocol for the anti-Markovnikov addition of thiols to alkenes and ring opening of epoxides by thiols catalysed by Amberlyst-15© in toluene under milder reaction conditions. Under same reaction conditions

Table 5
Amberlyst-15© catalysed epoxides ring opening by various thiols.[a]

Entry	Thiols	Product	Time (h)	Yield[b](%)
Reactions of 2-phenyloxirane with various thiols				
1			4	92
2			5	88
3			4	91
4			4	83
Reactions of 1,2-epoxycyclohexane with various thiols				
5			4	91
6			4	83
7			4	82
8			5	84
Reactions of epichlorohydrin with various thiols				
9			6	95
10			5	83
Reactions of 2-phenyloxirane with various thiols				
11			12	86
12			12	84
13			12	85
14			12	80
Reactions of 1,2-epoxycyclohexane with various thiols				
15			12	86
16			12	80

[a] Reaction conditions: epoxide (0.5 mmol), thiol (0.6 mmol), Amberlyst-15© (30 wt%), toluene (2 mL), temperature (80 °C).
[b] Isolated yield.

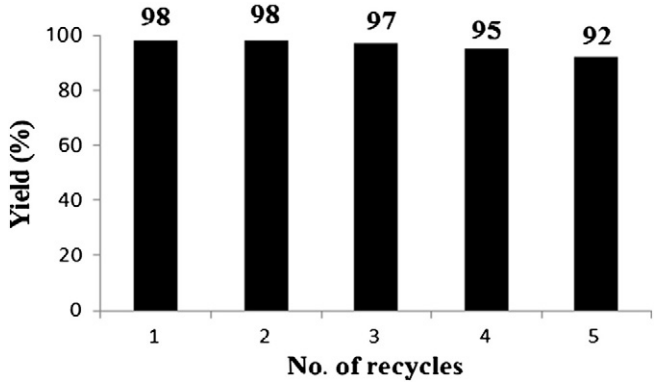


Fig. 1. Recyclability study of Amberlyst-15© catalyst: (a) Reaction conditions: styrene (0.5 mmol), thiophenol (2.0 mmol), Amberlyst-15© (30 wt%), toluene (2 ml), temperature (80 °C). Yield based on GC analysis.

dehydration of β-hydroxysulphides gives one pot synthesis of phenyl(styryl)sulfanes with excellent yields. The advantage of the metal free protocol includes inexpensive, safe to handle, commercially available catalyst, simple catalyst product separation so easy to recycle, very short reaction time and high yields of desired products. The study revealed that the recovered catalyst could be reused for five consecutive cycles, without loss in activity affording excellent yield of desired anti-Markovnikov addition product. As per environmental concerns the heterogeneous recyclable catalysts make methodology environmentally friendly and relevant to large scale industrial operations.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at http://dx.doi.org/10.1016/j.catcom.2013.06.032.

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