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Cerium catalyst promoted C–S cross-coupling: synthesis of thioethers, dapsone and RN-18 precursors†

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In this work, we present a novel, efficient and green methodology for the synthesis of thioethers by the C–S cross-coupling reaction with the assistance of $[Ce(L\text{-}Pro)_2]_2Ox$ as a heterogeneous catalyst in good to excellent yields. A scale-up of the protocol was explored using an unpublished methodology for the synthesis of a dapsone-precursor, which proved to be very effective over a short time. The catalyst $[Ce(L\text{-}Pro)_2]_2Ox$ was recovered and it was shown to be effective for five more reaction cycles.

Introduction

Cross-coupling reactions are considered to be a powerful strategy for the formation of carbon–carbon and carbon–heteroatom bonds.[1] This type of reaction has dramatically changed the way that organic chemists build a wide variety of organic compounds.[2] However, to perform a cross-coupling reaction, it is necessary to have an organometallic compound and an organic electrophile (halides and pseudo halides compounds) in the presence of a catalyst containing palladium,[3] nickel[4] or copper.[5] Since the first publication about the cross-coupling reaction a lot of improvements have been made over time, in particular that nucleophiles[6] participate well as catalysts. That is why several efficient catalytic systems have been developed for cross-coupling reactions.[7] Despite the elegance of the cross-coupling reaction, and owing to the wide application of different nucleophiles such as chalcogenides (ROH, RSH and RSe), some drawbacks existed. Specifically, for C–S cross-coupling, until recently, the construction of C–S bonds using a catalyst containing transition-metals (*e.g.* Pd, Ni and *etc.*) remained relatively rare compared to the other nucleophiles. It is known that sulfur species such as thiols and disulfides (RSH and R_2S_2) can poison a catalyst, deactivating the catalyst and irreversibly limiting the reaction.[2]

On the other hand, in recent decades the biological and medicinal properties of organochalcogen compounds have been studied owing to their antioxidant, antimicrobial, antitumor, anti-inflammatory, and antiviral properties.[8] Thioethers belong to an important class of compounds that are the building blocks of biological and therapeutic molecules.[9] Typically the preparation of thioethers involves the coupling of organic halides with thiols or disulfides.[10] Owing to the importance of this reaction in organic procedures, the transition metal-catalysed carbon–heteroatom bond has been receiving more attention in recent years.[11]

C–S bonds are present in drug-molecules across many health areas, such as HIV,[12] skin disease (*e.g.* leprosy)[13] and Alzheimer's disease.[14] Owing to this, and focusing on the previously mentioned information about C–S bond formation, it is necessary to develop less aggressive procedures, as well as more profitable, low cost and efficient methodologies.[15] The first report published on C–S cross-coupling was by Migita[16] and co-workers using tetrakis(triphenylphosphine)palladium for the preparation of aryl sulfides. Recently, Nejat *et al.* have designed recyclable catalysts for the formation of thioethers *via* C–S cross-coupling.[17] The improvements reported by Migita *et al.* highlighted palladium catalysts. However, palladium, iridium, rhodium, and ruthenium are costly transition metals and are generally toxic.[18] However, in contrast to the 4d and 5d catalysts, Nageswar[19] and co-workers reported the first protocol for C–S cross-coupling using lanthanides. In this procedure, the authors described the synthesis of thioethers using aryl halides, thiols and lanthanum(III) oxide, as a heterogeneous catalyst and they proved that it was possible to recover

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†Electronic supplementary information (ESI) available: Experimental procedures, spectroscopic details regarding MEV and EDS analysis (gold metallization, aluminum base metallization and without metallization), and all the spectra (^1H and ^{13}C NMR) for the products. See DOI: 10.1039/c9ob02171j

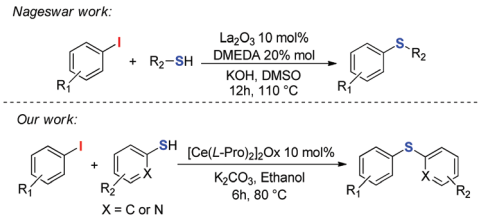


Fig. 1 Strategies for C–S cross-coupling using lanthanides as recoverable catalysts.

and reuse the catalyst. Motivated by Nageswar and co-author's work on lanthanides, we aimed to perform the C–S cross-coupling reaction using a cost-effective, easy to handle, and reusable cerium(III) catalyst. In 2016 our research group designed a cerium(III) heterogeneous catalyst which was applied in various procedures such as the Kabachnik–Fields reaction,[20] pyrazoles synthesis[21] and a one-pot 2,3-dihydroquinazolin-4(1*H*)-ones synthesis.[22] In a continuation of our work, here we report an efficient, cost-effective, low cost, short reaction time, green procedure and scale-up of Migita's reaction using thiols and aryl halides and applying $[Ce(L\text{-}Pro)_2]_2Ox$ as a heterogeneous catalyst (Fig. 1), as well as the synthesis of the bioactive compound RN-18 and a dapsone precursor.[23]

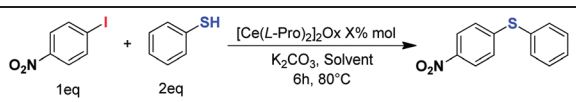
Results and discussion

Initially, we performed the C–S cross-coupling using a similar methodology to that described by Domingues and co-workers,[23] selecting a pattern reaction involving 1-iodo-4-nitrobenzene and thiophenol in ethanol. The first evaluation was performed without a catalyst (blank reaction entry #1, Table 1) and we only observed a 30% yield for the compound of interest. Furthermore, the same reaction was carried out using 5 mol% of the cerium catalyst (entry 2, Table 1) over 6 h in ethanol and resulted in 80% of the product (4-nitrophenyl)(phenyl)sulfane. After this, we performed the reaction using 7.5 mol% (entry 3, Table 1) and we obtained a 90% yield. To improve the yield for thioethers, we increased the cerium catalyst up to 10 mol% (entry 5, Table 1) which afforded a yield of more than 99%. Additionally, we performed a solvent assessment employing solvents such as water and toluene and we concluded that the reaction was compatible with polar solvents furnishing the best yields (entries 4 and 5, Table 1). To our delight, the methodology established by us, in comparison with others,[24] presented some important advantages like the use of green and low cost solvent such as EtOH, and the possibility of using an open flask reaction. Many reports in the literature reported the C–S cross-coupling reaction using an inert atmosphere owing to catalyst instability. Herein, the catalyst proved to be an air-stable catalyst and furthermore, was reusable. Both properties are important for the industrial application of the process.

With this valuable data in hand, we tried to decrease the amount of thiol in the C–S cross-coupling reaction to make the procedure even more practicable (Table 2). Interestingly, when the pattern reaction was performed using 1 equivalent of benzenethiol, we observed a decrease of the yield in the C–S cross-coupling (40%, entry 1, Table 2). However, when we performed the reaction using 1.5 equivalents of thiol, we observed an increase in the yield (78% yield, entry 2, Table 2). Notably, when we conducted the reaction using 2 equivalents of benzenethiol, we achieved an excellent yield of >99% (entry 3, Table 2).

It is important to note that the standard process affords the compound in a reduced time compared to many other reports.[25] Again, the above described catalyst efficiency in the C–S cross-coupling should be noted. To assess the scope for the synthesis of thioethers using this method and standard conditions we extended the protocol to different aryl halides and thiophenols (Table 3). The presence of electron-donating groups on the phenols (entries 3–9, Table 3) furnished excellent yields 71 → 99%. On the other hand, thiophenols with electron-withdrawing groups (entries 10–13, Table 3) afforded lower yields 31–82%. However, for thiosalicylic acid (entries 15 and 16, Table 3) we obtained excellent yields >99 and 86%, respectively. Regarding entry 15, a RN-18 precursor, a novel and potent Vif antagonist for HIV treatment[26] was produced in a higher yield, which is an improvement for RN-18 synthesis. With the change of substrates on the aryl halide (entries 17–19, Table 3) moderate tolerance of substituents was observed. To demonstrate once more the efficiency and appli-

Table 1 Optimization of reaction conditions[a]



Entry	Catalyst	Solvent	Yield[b]
1	—	EtOH	30%
2	5	EtOH	80%
3	7.5	EtOH	90%
4	10	EtOH	>99%
5	10	H_2O	91%
6	10	Toluene	35%

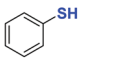
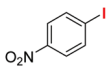
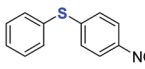
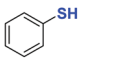
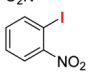
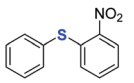
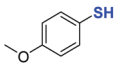
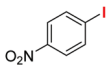
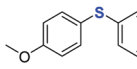
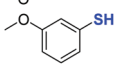
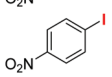
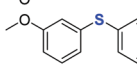
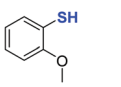
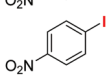
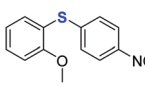
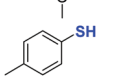
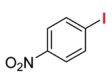
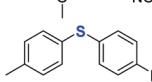
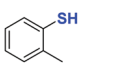
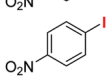
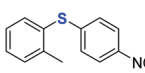
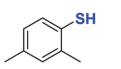
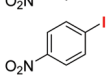
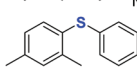
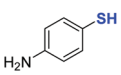
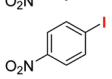
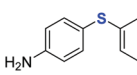
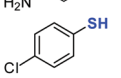
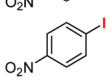
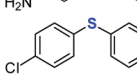
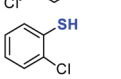
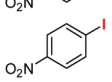
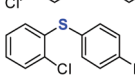
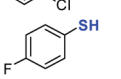
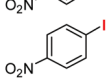
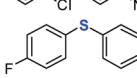
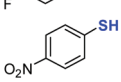
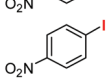
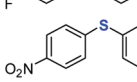
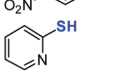
[a] Reaction conditions: 1-Iodo-4-nitrobenzene (0.5 mmol), benzenethiol (1.0 mmol), K_2CO_3 (2.5 mmol), and 4 mL of solvent. [b] Isolated yields using column chromatography.

Table 2 Study of the amount of thiol in the C–S cross-coupling reaction[a]

Entry	Thiol (mmol)	Yield[b]
1	0.5	40%
2	0.75	78%
3	1.0	>99%

[a] Reaction conditions: 1-Iodo-4-nitrobenzene (0.5 mmol), benzenethiol (*x* mmol), K_2CO_3 (2.5 mmol), and $[Ce(L\text{-}Pro)_2]_2Ox$ (10 mol%) in EtOH (4 mL). [b] Isolated yields using column chromatography.

Table 3 Scope of the C–S cross-coupling of thiols with aryl halides.[a]

Entry	Thiol	Aryl hallide	Product	Yield[a] (%)
1				>99
2				90
3				97
4				71
5				80
6				>99
7				88
8				89
9				96
10				82
11				75
12				33
13				31
14				65
15				>99
16				86
17				36
18				55
19				15

[a] Reaction conditions: Aryl halide (0.5 mmol), thiol (1.0 mmol), K_2CO_3 (2.5 mmol), and 4 mL of ethanol, 6 h, at 80 °C.

cability of the catalyst [Ce(L-Pro)$_2$]$_2$Ox we proposed the synthesis of an intermediary of the bioactive compound dapsone (Fig. 2).

For the Dapsone precursor (shown in Table 3, entry 6), the reaction was conducted using 4-aminothiophenol and 1-iodo-4-nitrobenzene affording a 96% yield. The same reaction involving a dapsone-precursor was carried out at a multi-gram scale

Fig. 2 The structure of the bioactive compound dapsone, for the treatment of skin disease.

(5 mmol) affording an 83% yield (1.02 g), in 9 h. In this procedure, with an increase in the molar equivalent of the starting materials (from 6 to 9 h), the procedure was completed in comparatively less time, indicating that this procedure can be easily applied in industry (Scheme 1).

Furthermore, the recyclability of the catalyst was studied (entry 1, Table 3) and it was found to be active in several cycles without exhibiting any significant loss for up to five cycles (Table 4).

We also found an increase in the catalyst weight per cycle (see Table 4). To explain the catalyst mass increment, we performed scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) analysis (Fig. 3 and 4) on the pre and post used catalyst.

A good similarity was observed between the SEM and EDS analysis of the pre (Fig. 3a and b) and post-reaction samples

Scheme 1 Gram-scale synthesis of a dapsone precursor using [Ce(L-Pro)$_2$]$_2$Ox.

Table 4 Recycling studies of [Ce(L-Pro)$_2$]$_2$Ox[a]

Cycle (#)	Yield[b] (%)	Catalyst recovery (%)
1	>99	100 + (58)
2	90	100 + (48)
3	86	100 + (45)
4	81	100 + (38)
5	76	100 + (24)

[a] Reaction conditions: 1-Iodo-4-nitrobenzene (0.5 mmol), benzenethiol (1.0 mmol), and K_2CO_3 (2.5 mmol) in EtOH (4 mL). [b] Isolated yield using column chromatography.

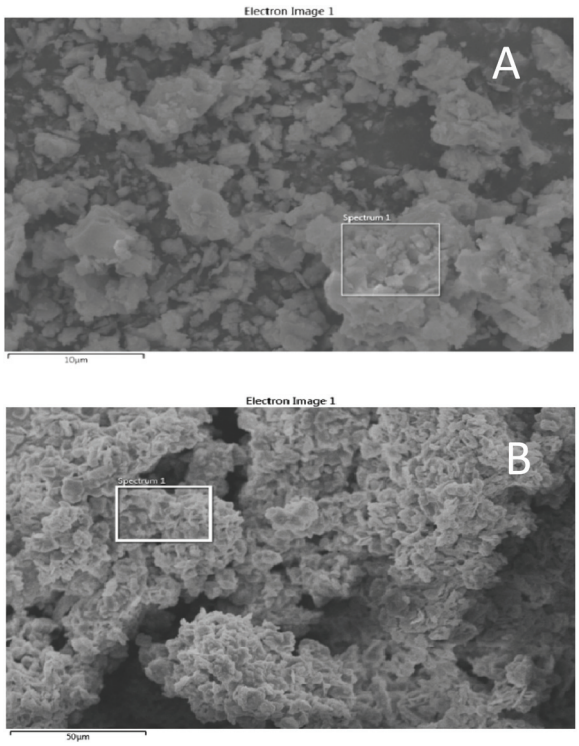


Fig. 3 SEM image of $[Ce(L\text{-}Pro)_2]_2Ox$ before the reaction (a) and after five cycles (b).

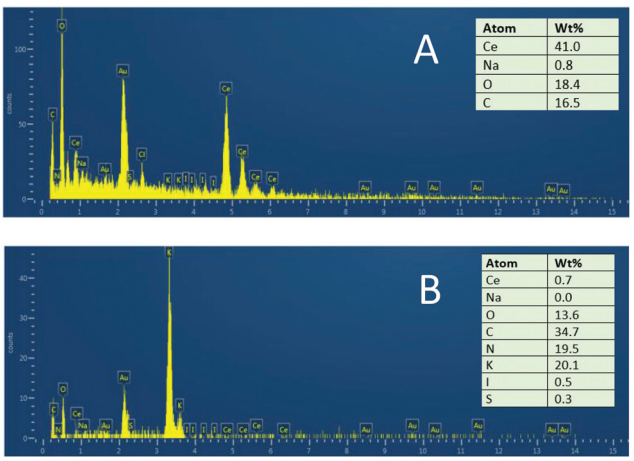


Fig. 4 EDS for $[Ce(L\text{-}Pro)_2]_2Ox$ before the reaction (A) and after five cycles (B).

(Fig. 3b and 4b), performed using gold based metallization. The EDS analysis revealed a difference between the pre and post-reaction catalyst in the atomic percentage of cerium, carbon, oxygen, and sodium.

This remarkable difference is due to the mechanism of the C–S cross-coupling, driven by $[Ce(L\text{-}Pro)_2]_2Ox$, affording a new complex demonstrated by the insertion of sulfur and aryl halide which strongly indicates a C–S cross-coupling catalytic cycle has occurred (see spectroscopy data in the ESI†).

Experimental

All chemical reagents and solvents were used without any specific treatment. The respective reactions were monitored by thin layer chromatography (TLC) MACHEREY-NAGEL (SIL G/UV_{254}). The purification of the compounds was performed by column chromatography on silica gel using appropriate quantities of hexane and acetyl acetate. ^1H and ^{13}C NMR spectra were recorded in $CDCl_3$ or DMSO on a Bruker (300 MHz and 75 MHz respectively) spectrometer.

Synthesis of the $[Ce(L\text{-}Pro)_2]_2Ox$ catalyst

$[Ce(L\text{-}Pro)_2]_2Ox$ was obtained using proline (5.0 mmol) in methanol (15 mL) and aqueous sodium hydroxide solution (5.0 mmol in 3 mL) at room temperature for 10 min. $CeCl_3{\cdot}7H_2O$ (2.5 mmol in 1 mL), was added to the reaction, and the solution was left stirring overnight. Sodium oxalate (0.1 g mL^{-1}) was used as a precipitating agent. The solid was filtrated off, washed with methanol and dried (92%).

General procedure for the C–S cross-coupling of thiols with aryl halides

Aryl halide (0.5 mmol) and thiol (1.0 mmol) were placed in a tube with ethanol (4 mL), K_2CO_3 (2.5 mmol) was added in the presence of 10 mol% of cerium catalyst, and the mixture was heated at 80 °C for 6 h. TLC was used to monitor the progress of the reaction. After the reaction was completed, the catalyst was filtered off and the solvent was removed under a vacuum. The residue was extracted with chloroform (2 × 20 mL) and dried over $MgSO_4$. The solvent was removed under vacuum and the crude product was purified using column chromatography on silica gel with appropriate quantities of hexane and ethyl acetate.

C–S cross-coupling of thiosalicylic acid with aryl halides (entries 15 and 16)

The cerium catalyst (10 mol%), aryl halide (0.5 mmol) and thiosalicylic acid (1.0 mmol) were placed in a tube and K_2CO_3 (2.5 mmol) in ethanol (4 mL) was added, the mixture was heated at 80 °C for 6 h. The progress of the reaction was monitored using TLC (EtOAc : hexane, 1 : 1). After the reaction was completed, the catalyst was filtered off, the solvent was removed under vacuum and then ice water was added to the reaction mixture, followed by acidification using 5 N HCl to obtain the crude product. The solid was filtered off and washed several times with petroleum ether and recrystallized using ethanol to afford the product.

C–S cross-coupling of thiols with 2-iodobenzoic acid (entries 17 and 18)

The cerium catalyst (10 mol%), 2-iodobenzoic acid (0.5 mmol) and the thiols (1.0 mmol) were placed in a tube and K_2CO_3 (2.5 mmol) in ethanol (4 mL) was added, the mixture was heated at 80 °C for 6 h. The progress of the reaction was monitored using TLC (EtOAc : hexane, 1 : 1). After the reaction was complete, the catalyst was filtered off, the solvent was removed

under vacuum and then ice water was added to the reaction mixture, followed by acidification using 5 N HCl to obtain the crude product. The solid was filtered off and washed several times with petroleum ether and recrystallized using ethanol to afford the product.

General procedure for the synthesis of the dapsone-precursor

Synthesis of the dapsone-precursor was carried out in the gram-scale and the reaction was performed using 1-iodo-4-nitrobenzene (5 mmol) and 4-aminothiophenol (10 mmol) in ethanol (4 mL), K_2CO_3 (25 mmol) was added in the presence of 10 mol% of cerium catalyst, and the mixture was heated at 80 °C for 9 h. TLC was used to monitor the progress of the reaction. After the reaction was complete, the catalyst was filtered off and the solvent was removed under vacuum. The residue was extracted with chloroform (2 × 20 mL) and dried over $MgSO_4$. The solvent was removed under vacuum to give the crude products, which were purified using column chromatography on silica gel using appropriate quantities of hexane and acetyl acetate (83% yield, 1.02 g).

Conclusions

In conclusion, $[Ce(L\text{-}Pro)_2]_2Ox$ was proved to be a highly efficient reusable catalyst for the synthesis of thioethers and derivatives in moderate to excellent yields under green conditions. It was possible to reuse the catalyst for five catalytic cycles. In addition, this methodology can be applied for the synthesis of two bioactive compounds, a dapsone precursor (entry 9, Table 3) (in scale-up) and RN-18 (entry 15, Table 3), which means that this methodology is applicable in the pharmaceutical industry.

Conflicts of interest

There are no conflicts to declare.

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