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Synthesis of Indenes via Graphene Oxide Mediated Manipulation of Morita-Baylis-Hillman Alcohols

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A new metal-free synthetic approach to functionalized indenes is documented. The use of commercially available graphene oxide (GO) allowed the direct access to indenyl cores (yield up to 80%) via intramolecular Friedel-Crafts-type allylic alkylations with readily available Morita-Baylis-Hillman alcohols. Combined experimental and spectroscopic investigations contributed to shed light on the reaction mechanism dealing with a nanostructured carbon material-based C–C bond forming reaction.

Introduction

1*H*-Indenes are a widely diffused organic scaffold in naturally occurring compounds as well as in synthetic medical ingredients with diversified pharmaceutical activities.[1] In addition, indenes find numerous applications in the realization of functional materials and as organic ligands for metallocene compounds.[2]

In this regard, the development of direct and efficient synthetic methodologies for their preparation attracted much attention within the chemical community (Figure 1a).[3] Among the known synthetic strategies directed to the preparation of functionalized indenes, intramolecular transition metal catalyzed cross-coupling condensation and cyclization reactions achieved great popularity due to consolidated modulability and site-selectivity.[4] Despite their efficiency, the need for densely functionalized aromatic precursors and the use of expensive metal catalysts represent important shortcomings towards a capillary diffusion of these methodologies on large scale productions.

With the aim of addressing cost efficiency, we took advantage of our recent discoveries on carbocatalysis[5,6] and (electrochemical) manipulation of Morita-Baylis-Hillman acetates.[7] In particular, the efficient electrophilic activation of π-alcohols (allylic and propargylic)[8a–b] and cyclobutanols[9] exerted by graphene oxide (GO) led us to propose readily achievable Morita-Baylis-Hillman alcohols as valuable precursors of indene-2-carboxylates **2** under the assistance of GO (Figure 1b).[10]

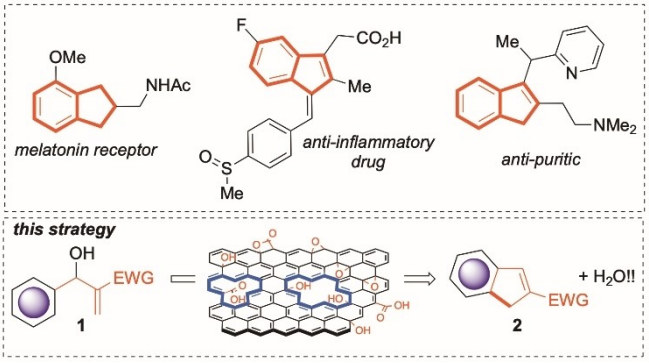

Figure 1. a) Examples of bioactive compounds featuring the indenyl core. b) The present GO based synthetic strategy to indenes via intramolecular Friedel-Crafts type intramolecular allylic alkylation with MBH alcohols.

It is worth mentioning that some Brønsted acid catalyzed/assisted synthesis of indenes from alcohols have been reported so far,[10] however, most of these examples rely on the pre-synthesis of elaborated allylic or propargylic alcohols, stoichiometric amounts of promoters and harsh conditions. Therefore, the seek for metal-free strategies for the preparation of indenes from readily available, simple, and yet highly modulable starting materials is highly desirable.[11]

Results and Discussion

At the outset of the investigation, we tackled the intramolecular FC-type alkylation on MBH alcohol **1a** as a model substrate.[12] Here, a wide range of reaction parameters were screened, with particular focus on nature and loading of the promoting agent,

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reaction temperatures, concentration and solvents. A complete list of results can be found in the supporting information file.

Satisfyingly, the use of commercially available GO (150 wt%) in refluxing DCE (80 °C) triggered the formation of indene **2 a** in 69 % yield over 16 h reaction time. The former turned out as the optimal conditions, and any variation led to a drop in chemical yield (Table 1).

In details, changes on the reaction media stressed the superiority of halogenated solvents in the titled protocol ($CHCl_3$: 64 % yield, entry 1). As a matter of fact, dioxane, acetonitrile and mesitylene performed worst in terms of isolated yields (no reaction or traces of product, entries 2–4). The loading of GO turned out to impact crucially the process as well. Here, while higher loading did not modify the chemical outcome of the process (200 wt%: 61 %yields, entry 6), dropping the quantity of GO to 20 wt% resulted in the formation of **2 a** only in traces (entry 5). The latter evidence strongly supports a GO surface-mediated catalysis during the reaction pathway (*vide infra*).

The reaction time was then investigated, and a prolonged run of 48 h was carried out without significant improvements (59 % yield, entry 8). Therefore, aiming to assess the specificity of GO to promote ring-closing processes with **1 a**, a range of additives were tested (entries 9–12). Here, while nanostructured reduced graphene oxide proved completely inert in the transformation, common Brønsted acids (*i.e.*, AcOH and *p*TsOH) furnished a complex reaction mixture (*p*TsOH) or complete recovery of unreacted **1 a** (AcOH). Analogously, $Sc(OTf)_3$ promoted the substantial isomerization of the MBH alcohol **1 a** to the isomeric cinnamyl isomer **1 a'**. Moreover, complete halogenation towards **1a''** was observed when stochiometric quantities of $AlCl_3$ were utilized (entry 11). Finally, a lower concentration of the reaction mixture was also contemplated to minimize undesired intermolecular self-condensations ([**1 a**]: 0.033 M, entry 13). Unfortunately, no positive result was recorded in the final chemical outcome (46 % yield).

Table 1. Variations from optimal reaction conditions.[a]

Run	Promoter (wt %)	Variations from optimal	Yield [%] **2 a**[b]
1	GO (150)	$CHCl_3$	64
2	GO (150)	CH_3CN	NR
3	GO (150)	Dioxane	NR
4	GO (150)	mesitylene	5
5	GO (20)	–	traces
6	GO (200)	–	61
7	GO (150)	45 C°	traces
8	GO (150)	48 h	59
9	*r*GO (150)	–	traces
10	$Sc(OTf)_3$ (20 mol%)	–	traces
11	$AlCl_3$ (100 mol%)	–	traces[c]
12	AcOH (100 mol%)	–	NR
13	GO (150)	[**1 a**] = 0.033 M	46 %

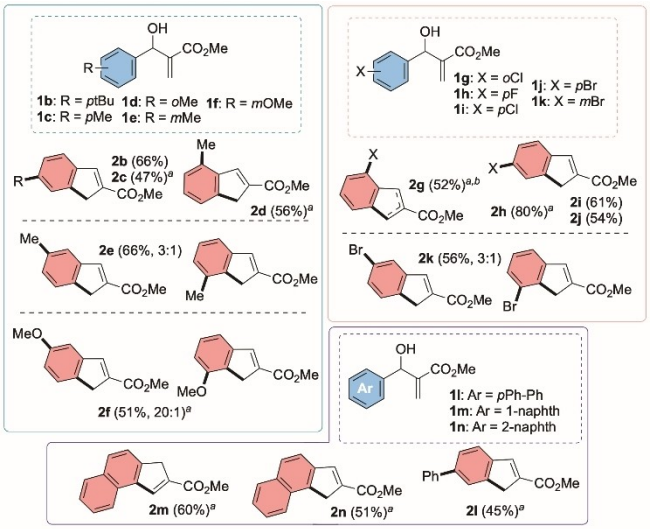
[a] All reactions were carried out under air in reagent grade solvents, unless otherwise specified ([**1 a**] = 0.1 M). [b] Isolated yields after flash chromatography. [c] The exclusive isomerization of **1 a** to the corresponding isomeric cinnamyl chloride **1a''** was observed. NR: no reactions.

With the optimal reaction conditions in hands, we assessed the generality of the protocol by subjecting to the GO-mediated ring-closing C–C bond forming reaction a range of Morita-Baylis-Hillman alcohols (**1 b**-**n**). The resulting chemical outcomes have been summarized in Scheme 1. In some cases, a slight deviation from optimal was applied to improve the reaction performance and reproducibility (GO 225 wt% and longer reaction times 48–72 h, see Scheme 1 caption for details).

First, it should be underlined that although a plausible shift of the C=C within the indenyl core could generate a mixture of indenes, in all cases (with the exception of **1 g**) only the indenyl core deriving from the formal intramolecular S_N2' allylic substitution was recorded.

Therefore, we accommodated electron donating groups (*i.e.*, Me, *t*Bu and OMe) at different positions (*ortho, meta* and *para*) of the aromatic ring. Interestingly, while the *para* and *ortho*-substituted precursors (**1 b**-**d,f**) delivered indenes **2 b**-**d,f** as single isomers carrying 6- and 4-substitution, respectively; the *m*-Me alcohol **1 e** led to a 3 : 1 mixture of indenes with an overall 66 % yield.

Then, the tolerance of the protocol toward substitutions with halogen atoms, was accounted. To this aim, F, Cl and Br atoms were arranged at different positions of the benzene unit (**1 g**-**k**). Moderate to very good yields were always obtained (up to 80 %) regardless the position of the substituent.



Scheme 1. Scope of the present methodology. Optimal conditions summarized in Scheme of Table 1 are utilized. [a] 225 wt% GO was employed (reaction times: 48–72 h, see supporting information for further details). [b] A 1 : 1 mixture of the two isomers was obtained.

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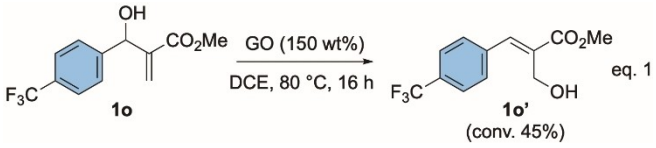
Also, in these cases the methodology proved high regioselectivity, and only in the case of the *m*-Br substituted precursor **1 k**, a 3 : 1 mixture of regioisomer (**2 k**, 66% overall yield) was isolated. Finally, oligoarenes-based indenes were also accessible through this strategy in moderate to good yields (up to 60%). In particular, *p*-phenyl substituted allylic alcohols **1 l** and naphthyl-based derivatives **1 m** and **1 n** reacted smoothly under optimal GO assistance. Interestingly, high chemoselectivity was recorded with MBH adducts **1 m,n** that performed the C–C ring-closure event at the C-1 position exclusively (51-60% yield).

Contrarily, the use of strongly deactivated aromatic compounds such as the CF_3-based arene **1 o** resulted inefficient in the cyclization, providing partial isomerization to the cinnamyl isomer **1 o'** (45% yield, Scheme 2).[13] This evidence prompted us to envision the preliminary alcohol isomerization as the initial stage of the whole process (*vide infra*).

With the aim of gaining more information on the elemental steps involved in the overall transformations, the model reaction **1 a** → **2 a** profile was monitored over time (initial 3.5 h) via NMR spectroscopy (Figure 2). Interestingly, an initial rapid formation of **1 a'** was recorded supporting the GO-assisted isomerization of the alcohol as the initial stage of the indene synthesis. Only after 1.5 h, the formation of significant amounts of indene **2 a** was observed, suggesting an initial inducing reaction time due to the formation of a critical concentration of cinnamyl alcohol **1 a'** as the real active species.



Scheme 2. Proving the initial isomerization of alcohols **1** by means of electron-deficient substrate **1 o**.

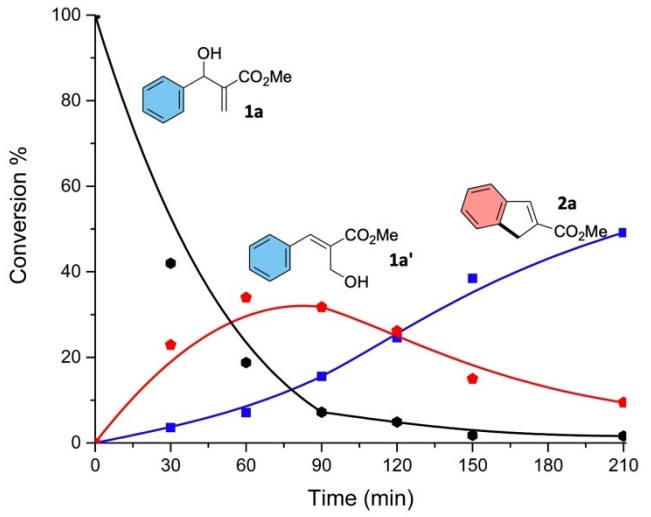


Figure 2. a) Monitoring of the GO mediated reaction profile over time (**1 a** → **2 a**). Reaction conversions were determined via ^{1}H-NMR analysis on the reaction crudes in the presence of mesytilene as the internal standard.

By keep subjecting the reaction mixture to reflux in the presence of GO, the formation of **2 a** increased further up to 49% (3.5 h) with the concomitant decrease of both alcohols **1 a** and **1 a'**. Additionally, the hot-filtration-test revealed a genuine GO-based mechanism and did rule out possible side activation processes promoted by Brønsted acidity or leaching phenomena (see SI).

Therefore, our focus moved toward a mechanistic interpretation with specific focus on the role of GO. First, insights were gained by analyzing the superficial morphology of the recovered GO material after the chemical transformation via XPS (C 1s signal Figure 3). The collected spectra revealed a net decrease of N, Cl and S contents on GO after catalysis, while O/C ratio decreased from 0.35 to 0.28. From C 1s fit (Figure 3) we observed the slight increase of C-OH (from 16% to 20%) and decrease of C–O-C (from 24% to 17%), that is usually associated with epoxy ring opening. This evidence: epoxy ring opening and decrease of oxidation – are compatible with a covalent anchoring of **1 a** on GO (epoxy ring opening) and with some **1 a** residual still present on GO after the catalysis, that might limit the number of active catalytic sites on GO (increase of carbon content due to **1 a** content and decrease of overall O/C).

Based on the interplay of experimental as well as spectroscopic evidence, and according to previous literature reports the mechanistic sketch depicted in Scheme 3 is proposed. In particular, isomerization of **1** to **1'** is initially postulated under both Brønsted acidity as well as GO catalysis.[14] Then, the GO surface can activate **1 a'** via interaction with the epoxide contents of the carbon lattice. Protonation of the epoxide (**A**) could favor the consequent ring-opening procedure by means of alcoholysis step.[15] The highly activated allylic ether **B** is thus

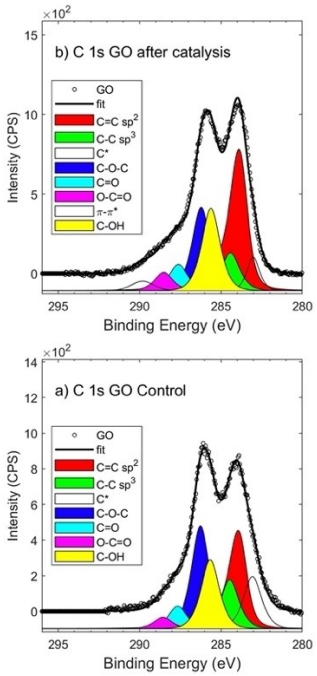


Figure 3. a) High resolution deconvoluted XPS spectra of C 1s peak of GO control (a) and recovered GO after catalysis (b). [GO control sample (80 °C in DCE for 16 h).]

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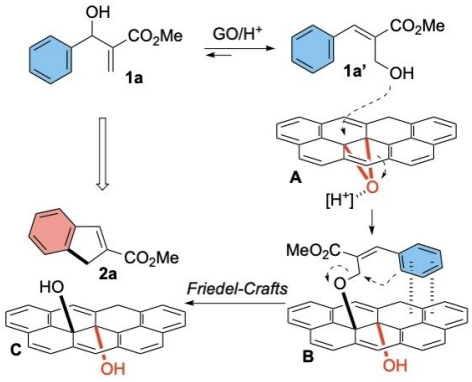

Scheme 3. Mechanistic sketch of the present synthesis of indenes.

formed; stabilizing π- π contacts among the π-content of the alcohol and the aromatic domain of the GO can also be invoked. The latter geometrical arrangement could trigger the final intramolecular Friedel-Crafts-type allylic alkylation promoting the formation of the indene **2 a** and releasing the modified GO **C**.

This mechanism would account for the almost net redox-neutral transformation regarding the GO (see XPS analysis) and for the detected conversion of part of the epoxide content into alcoholic groups. Moreover, the inertness of **1a''** toward the present C–C bond forming step supports the proposed initial mechanist stage involving the epoxide ring-opening event.

We finally accounted for the recovering and reutilization of the heterogeneous catalyst. The purification step of the reaction crude was facilitated by the insolubility of the GO in the reaction media, however all attempts to regenerate (acid and oxidative treatments) and reutilize the recovered GO in subsequent runs were unfruitful.[16] Although a conclusive explanation for the present evidence is still unknown we could speculate that some deep morphologic or aggregative changes could affect the GO during the catalytic run, preventing the active form of the material to be re-utilizable in subsequent attempts.

Conclusions

In conclusion, we have documented on the unprecedent efficiency of commercially available graphene oxide towards the synthesis of substituted indenes starting from readily available Baylis-Hillman alcohols. The heterogenous promoter addressed still unsolved synthetical issues, since common Lewis as well as Brønsted acids failed in triggering the ring-closing event, effectively. The mechanism of the process was also deeply investigated to shed light on the unique role of GO. Here, experimental, and spectroscopic investigations revealed a multiple activation mode exerted by GO, namely: acid catalyzed isomerization of the starting alcohol and subsequent covalent activation toward the final indene products. Studies towards the employment of the catalytic performance of GO towards organic transformations dealing with activated alcohols are still running in our laboratories and will be documented in due course.

Experimental Section

A sample vial was charged with 1 mL of reagent grade DCE, 0.1 mmol of **1 a** (18 mg) and 27 mg of GO (150 wt%). The solution was poured in a pre-heated oil batch (80 °C) and kept stirring at the same for 16 h. After cooling, the GO was removed via filtration on a plug of silica, the insoluble residue was washed with AcOEt, the organic phases collected, and the volatiles evaporated under vacuum. Then, the reaction crude was purified via flash chromatography (*n*Hex:AcOEt 20 : 1) giving **2 a** in 64 % yield as a withe solid.

Supporting Information

Additional references cited within the Supporting Information.[17–22]

Acknowledgements

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Conflict of Interests

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings will be available in IRIS at https://cris.unibo.it/ following an embargo from the date of publication to allow for commercialization of research findings.

Keywords: alcohols · carbocatalysis · graphene oxide · indenes · Morita-Baylis-Hillman

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[13] Unfortunately, at the present, we do not have an exhaustive explanation to account for the dichotomic behavior of substrates **1 h** and **1 o**. Perhaps, steric effects could result predominant in the case of the CF_3- with respect to the F-containing substrate, in the productive interaction of the GO with the respective MBH acetates **1** at the early-stage of the reaction machinery.

[14] The addition of K_2CO_3 (2 eq.) to the reaction mixture prevented the isomerization step, supporting the acid mediated mechanism. Although we cannot exclude that partial decomposition of DCE could generate serendipitous traces of HCl, it is known that dispersions/solutions of GO intrinsically generate acidic environments due to the multiple acid moieties present both at the edges and basal planes of the carbon nanomaterial

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