

Safer, Faster and Cleaner Reactions Using Encapsulated Metal Catalysts and Microwave Heating

PERFORMANCE ENHANCEMENT OF PALLADIUM, PLATINUM AND OSMIUM CATALYSTS

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The combination of focused microwave heating and encapsulated metal promoters (EnCat™) offers a safer, cleaner and more cost-effective solution to a wide range of catalyst-mediated reactions, some of which are not widely accessible to the bench chemist due to high hazard ratings. These include the palladium-catalysed Sonogashira cross-coupling, palladium-catalysed transfer hydrogenation, platinum-mediated hydrogenation and osmium tetroxide-catalysed dihydroxylation.

Microwave heating has developed as an important tool for research chemists, enabling reactions to be carried out and optimised more quickly than using traditional heating methods (1–3). Direct irradiation of the reaction mixture produces a more uniform and homogeneous heating profile than does, for example, an oil bath. In most cases the observed increase in rate can be explained by the extremely efficient energy transfer and homogeneous heating effect. This can lead to superheating of the reaction mixture (4): indeed, even microwave heating of an open vessel can achieve temperatures several degrees higher than the boiling point of the solvent (5).

In certain cases the presence of elements that strongly absorb microwave energy and release it efficiently as heat can cause localised ‘hotspots’ tens of degrees higher than the bulk temperature, generating significant rate enhancements (6–8). This effect can be exploited to heat materials of low microwave absorbance by the use of ‘passive heating elements’ (9). Non-polar and poorly absorbing solvents can also be superheated by adding small amounts of a strongly absorbing cosolvent such as an ionic liquid (10–13). The application of this selective heating can be particularly striking when the element is a heterogeneous

catalyst (14–16). A localised increase in temperature at a catalyst surface over the bulk temperature, or a selective absorption of microwave energy by catalytic species or organometallic intermediates on a reaction pathway, can lead to increased selectivity for the catalytic process while unwanted (thermally driven) side reactions are minimised by a relatively low bulk temperature (17). A synergistic advantage between microwave heating and platinum group metal catalysis can therefore be demonstrated (18).

The use of commercially available focused (monomode) microwave units (19–21) enhances the safety and reproducibility of reactions. The standard integration of monomode units into many laboratory environments has expanded the armoury of techniques available to chemists, allowing ready access to previously difficult-to-achieve chemistries. These include high-temperature reactions such as Ullmann couplings (22); some heterocycle preparations previously requiring metal baths (23, 24); the use of near-critical water as solvent (25–29); and shortening the reaction time on slow processes such as cycloadditions (30) to practically useful timescales, including replacing the need for autoclaves (31); and automated peptide synthesis (32, 33).

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For the reasons discussed, metal-catalysed reactions work particularly well under microwave irradiation; however safety and isolation issues still arise from their use. Elemental metal can deposit from reaction mixtures onto the side of the glass tube, causing localised superheating of the glass and explosive rupture of the vessel (34). This can occur with both homogeneous and heterogeneous catalysts. It can also be difficult to remove metal species selectively from the product on completion of the reaction.

The EnCat™ range of encapsulated metal catalysts were designed to address these issues of purification and reuse. Unlike other immobilised homogeneous catalysts such as FibreCat™, where phosphine ligands are attached to polyethylene fibres (35), the homogenous catalyst in EnCat is contained within a resin microcapsule. The use of such supported or ‘heterogenised’ catalysts industrially is being driven by regulatory pressures towards lower residual levels of metal catalysts within active pharmaceutical intermediates (APIs) (36, 37).

EnCats are prepared by an interfacial micropolymerisation of an organic solution containing the homogeneous metal catalyst, monomers (functionalised isocyanates) and additives, dispersed as a suspension in an aqueous phase. Reactive groups generated at the interface combine to form polymer walls and, as the surrounding matrix forms, the catalyst is entrapped to give spherical microcapsules (38). The individual catalytic species gain additional stabilisation through interaction with the amide functionality of the polyurea matrix, resulting in very low levels of metal leaching. Consequently the catalyst can be recovered efficiently by simple filtration and reused.

Examples of catalysts already encapsulated this way include palladium(II) acetate (39, 40) with and without various phosphine ligands (41), palladium(0) nanoparticles (42), platinum(0) (43) and osmium tetroxide (44). Here we describe how EnCats provide a homogeneous catalyst in a more effective form for use with microwave heating.

EnCats in Microwave Heated Reactions

EnCats have been shown to be highly compatible with microwave heating (45, 46). Following the excel-

lent work by Ley and coworkers in demonstrating microwave-enhanced palladium EnCat-catalysed Suzuki couplings in both batch and flow modes (47), we were keen to understand the role of EnCat in heating bulk solution. Ley found that cooling reactions while providing a fixed microwave power equivalent to that required for good conversion in the non-cooled method resulted in cleaner products at similar or better conversions. The lower bulk temperature in the case of cooling may explain the reduction in side reactions, with the temperature ‘inside’ the EnCat beads potentially much higher. It is known that Pd/C preferentially absorbs microwave energy when suspended in a virtually microwave-transparent solvent, and ‘passively’ heats the surroundings (48). To investigate whether EnCat acts in the same way, a 5 cm³ sample of anhydrous toluene, with various additives, was irradiated at a constant power of 200 W for 5 minutes and the temperature recorded (Figure 1). Adding 250 mg of Pd EnCat had a negligible effect on the heating profile, as did the addition of ‘blank’ EnCat beads containing no metal. Addition of an equivalent amount of homogeneous palladium acetate (27 mg) also had no effect on the heating behaviour, whereas 50 mg of palladium (5%) on carbon caused a significantly increased rate of heating.

These results suggest Pd EnCat does not cause superheating of the bulk solution, and behaves more like homogeneous palladium acetate than palladium on carbon.

Palladium(II) for Cross-Coupling Reactions

Considerable effort has been focused on the use of Pd EnCat to facilitate cross-coupling reactions (41). The extremely low leaching of metal species and ease of handling of EnCat beads greatly simplify purification of these reactions. Many examples have been published regarding the use of EnCats with microwave heating for the acceleration of specific reactions (49–52). An important advantage, often not considered, is improved safety when using EnCats in a microwave reactor. Deposition of a film of elemental metal on the glass walls of microwave tubes by precipitation from solution is a common problem with conventional metal catalysts. This has been shown not to occur with Pd EnCat (53). Where a

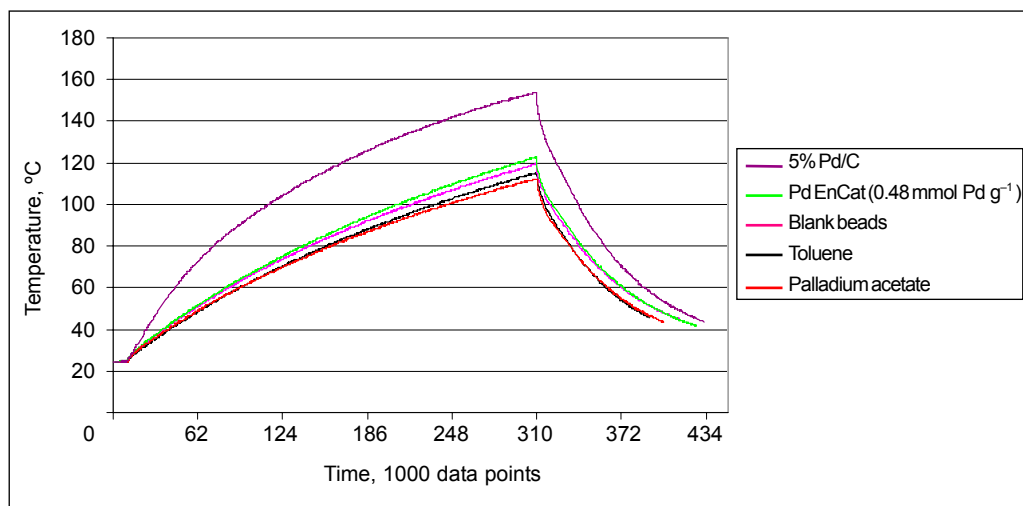


Fig. 1 Rate of heating of toluene containing various dopants under microwave irradiation

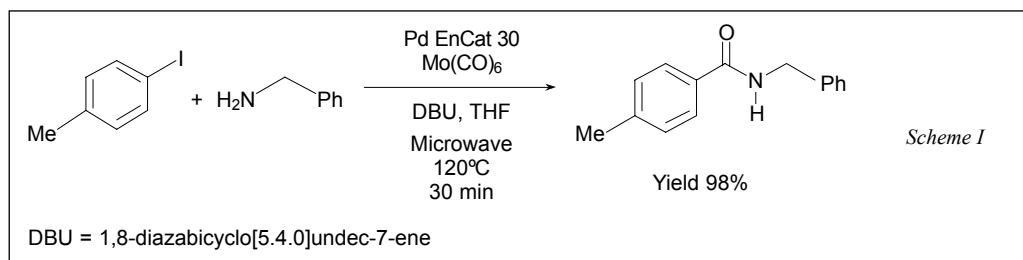
film is deposited, it absorbs microwave energy strongly, and hotspots can form, resulting in vessel failure. With modern microwave reactor designs such ruptures are well contained; however the release of vapours and subsequent decontamination pose serious issues. These can lead to restrictions on the use of particularly hazardous reagents.

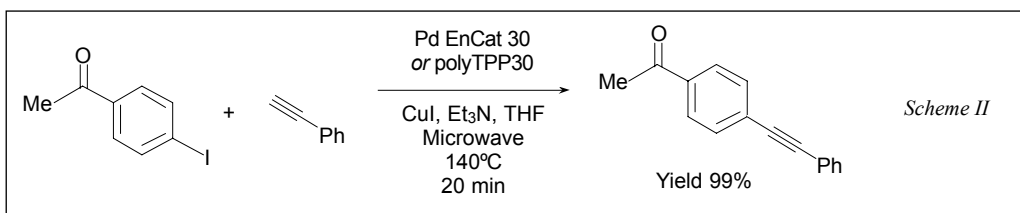
By way of example, a useful palladium-mediated microwave process is the carbonylation of aryl halides with solid sources of carbon monoxide (54). Molybdenum hexacarbonyl has been shown to be an effective carbon monoxide releasing agent (55, 56), however it is a very toxic substance with relatively high volatility (57). The risk of vessel rupture in such procedures can be greatly reduced by substituting Pd EnCat for the traditional palladium catalyst. The reaction proceeds with quantitative conversion as shown in Scheme I.

EnCats have been applied in flow chemistry with the beads packed in simple columns and

reagents passed over them. The initial work in this area is extremely promising for the process-intensification of homogeneous catalytic reactions (47, 58–60).

A low degree of leaching of the catalytic species is vital in a continuous process, in order to avoid rapid deactivation and resulting contamination of the product flow stream. Certain substrates are known to induce leaching of palladium from EnCat resins, with aryl iodides and alkynes showing a high propensity. Indeed, running the microwave-assisted Sonogashira reaction in Scheme II with Pd EnCat 30 resulted in product with a palladium content of 83 ppm. The triphenylphosphine-entrapped Pd EnCat (polyTPP30) resin demonstrates an extremely high retention of both the palladium and phosphorus ligand, and has been used to great effect in the same reaction (Scheme II). Using Pd EnCat polyTPP30 as the catalyst, the residual palladium concentration in the product was only 14 ppm.





Palladium(0) for Hydrogenation Reactions

The nanoparticulate palladium(0) EnCat catalyst has been demonstrated as a highly chemoselective hydrogenation and transfer hydrogenation catalyst (61, 62). In addition to the improved selectivity shown by Pd EnCat NP30, a superior safety profile and ease of handling make it a powerful alternative to palladium on charcoal.

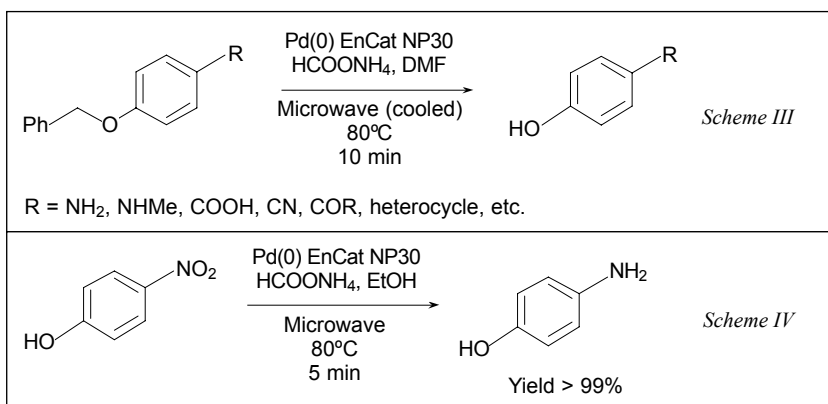
Transfer hydrogenation with Pd EnCat NP30 is easily performed in the microwave, allowing reactions in minutes rather than hours. A recent paper by Quai and coworkers demonstrated the efficiency of microwave-assisted transfer hydrogenation for *O*-benzyl deprotection (Scheme III) (63). The use of EnCat was recommended to improve the safety of the process and reduce palladium contamination of the products.

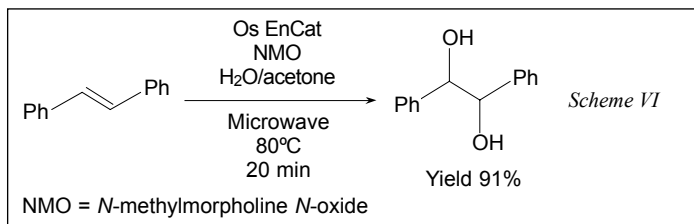
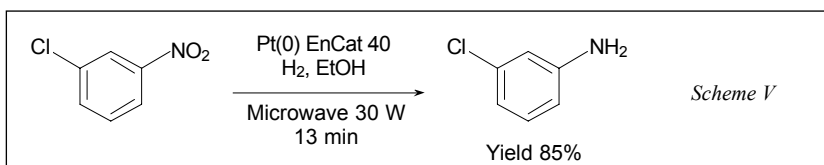
Scheme IV shows a representative example of an aromatic nitro reduction. These reactions are conventionally carried out at ambient temperature overnight (64). However, the microwave transfer hydrogenation procedure gave a quantitative conversion to the final product in only 5 minutes.

Platinum(0) for Hydrogenation and Reduction Reactions

To complement the palladium(0) EnCat range, a platinum(0) EnCat has recently been developed, offering the same benefits over its carbon-supported equivalents as the palladium version: improved safety profile, ease of handling and low metal leaching. Pt(0) EnCat 40 performs similarly to Pt/C in hydrogenation reactions, and is particularly useful in selective reductions in the presence of aryl chlorides. The reaction shown in Scheme V gave 3-chloroaniline with > 98% selectivity at room temperature under an atmosphere of hydrogen after one hour (65). Microwave-assisted hydrogenations have recently been investigated (66), and equipment to run them in the laboratory is becoming commercially available (67, 68). With microwave reactors designed to meter pressures up to 15 bar and run at them, such technology offers the bench chemist simple, safe access to hydrogenation.

The microwave-assisted hydrogenation of 3-chloronitrobenzene shown in Scheme V was run using a standard microwave vial. A hydrogen atmosphere (at slight positive pressure) was introduced *via* a needle and manifold cycled between vacuum and hydrogen from a lecture





bottle. Following irradiation at a constant power (30 W) for 13 minutes all the starting material was consumed, giving 3-chloroaniline in 85% yield. With equipment designed to charge gas to a given pressure and monitor the pressure drop, it is to be expected that this reaction could be optimised to higher selectivities.

Encapsulated Osmium Tetroxide for Dihydroxylation Reactions

The osmium tetroxide-catalysed dihydroxylation reaction is Nobel Prize-winning chemistry (69); however the routine use of osmium in the laboratory is avoided where possible due to its toxicity, the likelihood of contact due to its volatility and its propensity to cause burns (70). Os EnCat 40 is an encapsulated osmium tetroxide that is safer to handle because no osmium tetroxide vapour can escape the polymer matrix (44). The EnCat acts as a reservoir of osmium tetroxide, releasing catalytic amounts under oxidation reaction conditions, but retaining sufficient activity for recycling (71). Following the reaction only very low levels of residual osmium are detectable in the reaction media. Os EnCat 40 has been successfully applied to asymmetric dihydroxylation reactions (72). To demonstrate the application of Os EnCat 40 under microwave conditions, the simple dihydroxylation in Scheme VI was carried out at 80°C and was complete in 20 minutes. The corresponding reaction at ambient temperature, when allowed to proceed overnight, gave the product in 86% yield (73). With the reaction performed in a sealed microwave tube, the contents could be removed

via syringe with a fine filter fitting, minimising contact and potential hazards, and allowing routine, safe use of such chemistry.

Conclusions

Microwave heating has expanded the arsenal of synthetic methods available to the bench chemist. The use of encapsulated platinum group metal catalysts coupled with the inherently safe design of modern microwave apparatus enables safe access to an even greater range of useful transformations. Such a synergistic combination of technologies enables reactions to be performed that furnish clean products with very low levels of residual metal, thus simplifying the preparation of complex molecules.

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