

MINI REVIEW

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Nano-catalysts with magnetic core: sustainable options for greener synthesis

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Abstract

Author's perspective on nano-catalysts with magnetic core is summarized with recent work from his laboratory. Magnetically recyclable nano-catalysts and their use in benign media is an ideal blend for the development of sustainable methodologies in organic synthesis. Water or polyethylene glycol (PEG) provides good medium to perform such chemical reactions with magnetic nano-catalysts, as this combination adds exceptional value to the overall sustainable process development. In this mini-review, the uses of magnetically recyclable nano-catalysts for a variety of organic reactions are described in conjunction with activation via microwave irradiation.

Keywords: Greener synthesis, Nano-catalysis, Magnetic nano-catalysts, Microwave irradiation, Aqueous medium

Chemical developments in the new millennium are now routinely utilizing the concept of "green chemistry" to meet the challenges of protecting the environment and human health while maintaining commercial viability. *Green Chemistry* is defined as "the utilization of a set of principles that eliminates or reduces the use or generation of hazardous substances in the design, manufacture, and application of chemical products" and emphasizes hazard reduction as the performance criteria while designing new chemical processes. Nanotechnology processes in the recent years have enabled paradigm changing developments in environmental science, medicine and importantly, catalysis. One of the key areas for achieving sustainability is to explore the generation of efficient catalytic processes, via nano-catalysis [1] as a viable option; catalysis has been an integral part of what is now defined more clearly under nanotechnology domain [2].

Nanoparticles-Greener synthesis

Nanoparticles are the miniscule building blocks for an array of new commercial products and consumer materials in the emerging field of nanotechnology; they are discovered and introduced in the market place at a very fast pace. Nanoparticles can be defined as particulate matter with at least one dimension that is less than 100 nm. The

commercial interest in nanotechnology has significantly increased with more than US \$9 billion in investment from public and private sources [3]. Nanoparticles have an exceptionally large surface area to volume ratio, an important trait that is responsible for their widespread advantageous use in catalysis. The nanoscale size and shape imparts unique properties to catalysts because of the structural and electronic changes which differentiates them from the bulk materials. The fine tuning of nano-catalysts, in terms of composition (bimetallic, core-shell type or use of supports), shape and size has accomplished greater selectivity.

In the catalysis arena, the production of engineered nanomaterials is a major breakthrough in material science. A sustained effort has been made to develop eco-friendly strategies to generate these nanomaterials via pathways that use benign reagents rather than the hazardous substances conventionally used. The sustainable strategy for the preparation of nanoparticles has been exemplified by the use of vitamins B₁, B₂, C, and tea and wine polyphenols [4], which function both as reducing and capping agents [5]. This obviates the need to use toxic reducing agents, such as borohydrides or hydrazines. These extremely simple and aqueous green synthetic methods generate bulk quantities of nano-catalysts without the need for large amounts of insoluble templates [6] and have found numerous applications in catalysis [1,5,7].

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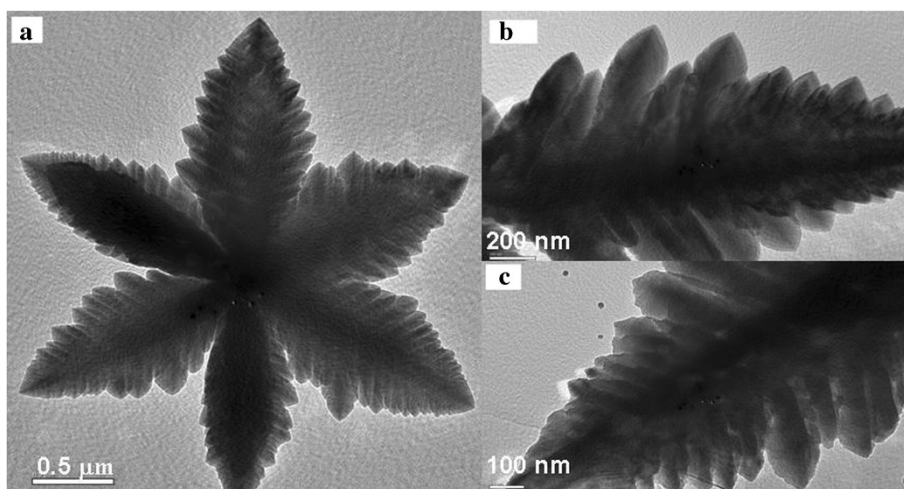
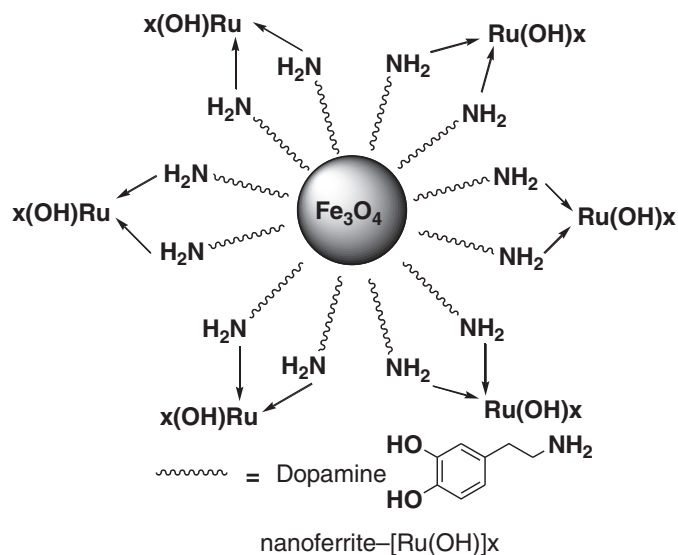
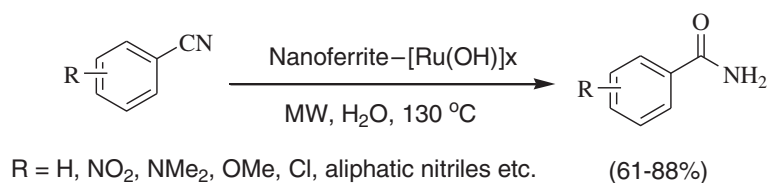


Figure 1 MW Synthesis of dendritic nano-ferrites (micro-pine morphology) from inexpensive starting materials in water without using any reducing or capping reagent agent; panels b and c show the well-defined and highly ordered branches. (Reproduced by permission from Royal Society of Chemistry, reference 7).

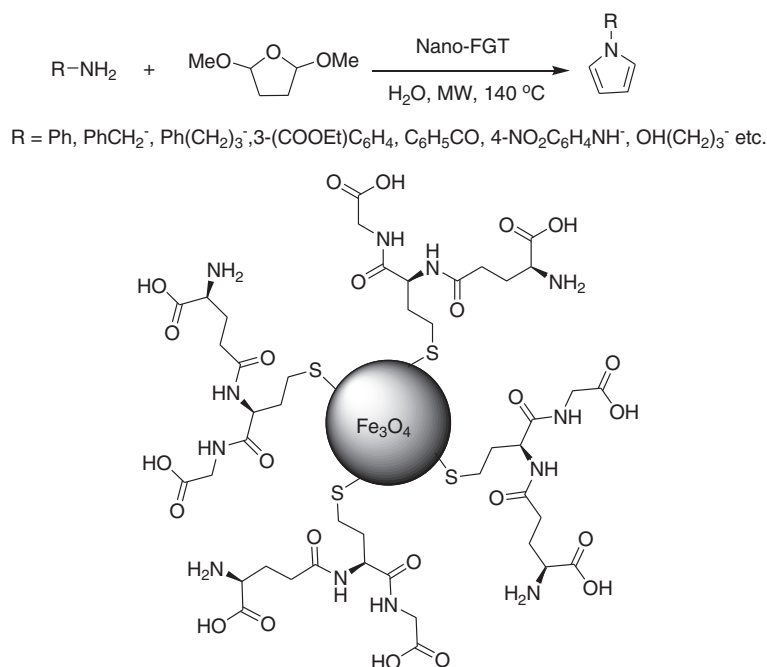
Alternate energy input- Use of microwave irradiation

The desired approach to the preparation of uniformly small-sized nano-catalysts may include alternative activation methodology, such as microwave (MW)-, and ultrasonic irradiation and mechanochemical mixing

[8]. The rapid and in-core MW heating has proven a useful method for the synthesis of these metallic nanostructures in solutions [9,10]. The valuable application of this method has been established for the preparation of silver (Ag), gold (Au), platinum (Pt), and gold-palladium (Au-Pd) nanostructures [5,11]. MW heating conditions



Scheme 1 Nanoferrite-[Ru(OH)]_x catalyzed aqueous hydration of nitriles (reproduced by permission from Royal Society of Chemistry, reference 7).



Scheme 2 Nano-FGT-catalyzed Paal-Knorr reactions (reproduced by permission from Royal Society of Chemistry, reference 7).

not only allow the rapid preparation of spherical nanoparticles, but also enables the formation of single crystalline polygonal plates, sheets, rods, wires, tubes, and dendrites as well [12]. Nanostructures of uniformly smaller sizes and with narrower size distributions and a higher degree of crystallization have been obtained using MW heating than those prepared via the conventional oil-bath heating; MW approach allows the greener synthesis of nanomaterials with several desirable features, such as shorter reaction times, better product yields and reduced energy consumption (Figure 1) [7].

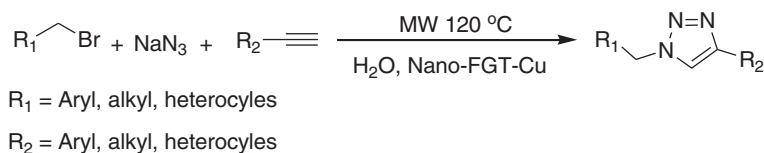
Importantly, the strategy encompasses “*benign by design*” principles and focus on the utilization of renewable resources, if possible [4,5].

Nanoparticles with magnetic core

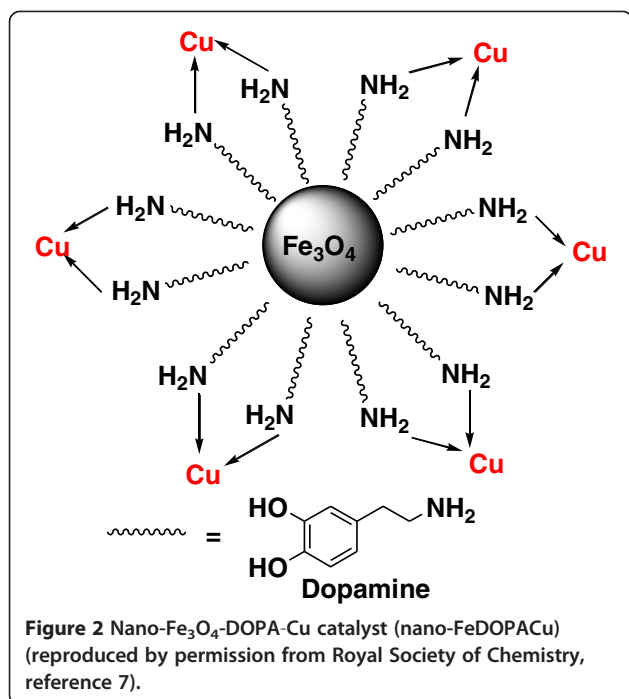
To protect the environment and to avoid undue manipulation efforts to purify the product and separation of the catalysts, there is a pressing need to develop methodologies that can facilitate recycle and reuse of these materials at

low concentrations and in complex matrices. In this context, iron-based magnetic nanoparticles (MNPs) have been developed that address these needs [13]. These MNPs can be further divided based on the nature of the magnetic core, which can be made up of either reduced species or oxides such as iron oxide NPs (Fe₂O₃ and Fe₃O₄) that has found application in oxidative and coupling reactions [14,15]. Such unmodified bare iron oxides comprising Fe₃O₄ and Fe₂O₃ are found to be active catalysts for the coupling of aldehyde, alkyne, and amine (A³ coupling) to afford an easy route to propargylamines [16].

The use of MNPs as catalysts in chemical synthesis has been extensively studied in recent years as the recovery of expensive catalysts after their use are some of the salient features in the sustainable process development [17-21]. MNPs coated with benign ligands such as dopamine or glutathione have also been developed and used as heterogeneous catalysts for numerous organic transformations and syntheses [17-21]. The functionalization of the surfaces of nano-sized magnetic materials provides a



Scheme 3 Nano-FGT-Cu catalyzed 1,3-dipolar cycloadditions reaction (reproduced by permission from Royal Society of Chemistry, reference 7).



quasi-homogeneous phase in reaction media and acts as a bridge between heterogeneous and homogeneous catalysis thus retaining the relative virtues of both of the systems.

The greener generation of nanoparticles and their eco-friendly applications in catalysis via magnetically recoverable and recyclable nano-catalysts for a variety of oxidation, reduction, and condensation reactions [20-25], has made a tremendous impact on the development of sustainable pathways. This heterogenization of the catalyst in the form of MNPs allows them to be recovered using an external magnet and facilitate their subsequent reuse more efficiently. In view of the reduced size of MNPs in nm range, most of the catalysts surface is accessible for reaction as it provides quasi homogeneous media for the catalysts.

Synthetic modification of magnetic nanoparticles

The synthetic modification of nanoferrites with dopamine and subsequent anchoring of the metal particles (Scheme 1) on its surface provides numerous opportunities to deploy these nano-catalysts effectively. The hydration of benzonitrile

with ruthenium hydroxide on magnetic nano-ferrites transforms it to benzamide in water is an example [22]. The catalyst can be easily separated using an external magnet and after its separation, the clear reaction mixture could be cooled to generate the crystals of benzamides. The complete procedure could be conducted exclusively in aqueous medium that is devoid of organic solvents [22]; the MW-assisted reaction proceeds with high turnover numbers and is attributed to the use of a nano-catalyst.

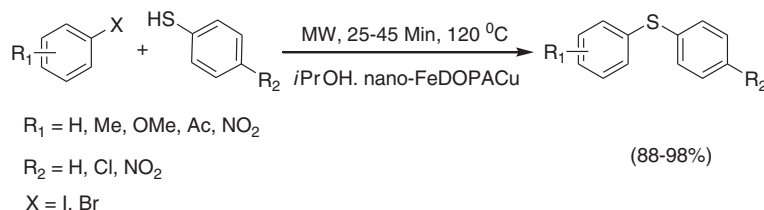
In an analogous manner, ubiquitous glutathione (GT) molecules have been immobilized on magnetic nanoferrites via their thiol groups [20] and the resulting catalyst (nano-FGT) has been used for the efficient synthesis of a wide variety of aryl, alkyl, and heterocyclic amines (Scheme 2). Such organocatalytic approach enables facile conversion of functionalized amines selectively into the corresponding pyrroles without affecting several sensitive functional groups [25]. This salient feature of immobilization of organocatalysts on MNPs enables their separation magnetically, after completion of the reaction, thus avoiding the cumbersome traditional separation by chromatographic means and solvent usage.

Such nanoparticle-supported and magnetically recoverable organocatalysts help catalyze the synthesis of heterocyclic entities (Paal-Knorr and other reactions) in pure aqueous medium thus precluding the use of toxic organic solvents, even in the work-up stages [1,7,25].

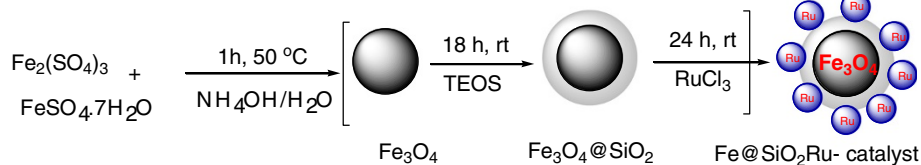
Magnetically separable nano-FGT-Cu catalyst could be used for azide alkyne cycloaddition (AAC) reaction and this general reaction is performed in one-pot via *in situ* azide generation followed by cycloaddition in aqueous media (Scheme 3) [26].

A similar nano-FeDOPACu bimetallic catalyst (Figure 2) has been deployed in the C-S coupling of aryl halides with thiophenols under MW irradiation conditions [27]; several iodides and bromides (with the exception of aryl chlorides) undergo reaction with thiophenols, affording the corresponding diaryl sulfides (Scheme 4).

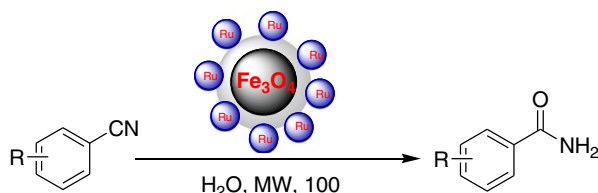
The activity of a Fe-Cu bimetallic catalyst can, however, be altered by changing the anchoring ligand for the immobilization of copper nanoparticles on magnetic nano ferrite (Fe₃O₄) surface. When glutathione is used, the catalyst shows activity for Huisgen cycloaddition [26].



Scheme 4 Nano-FeDOPACu catalyzed cross coupling of aryl halides with thiophenols.



One pot synthesis of nano-Fe@SiO₂Ru catalyst



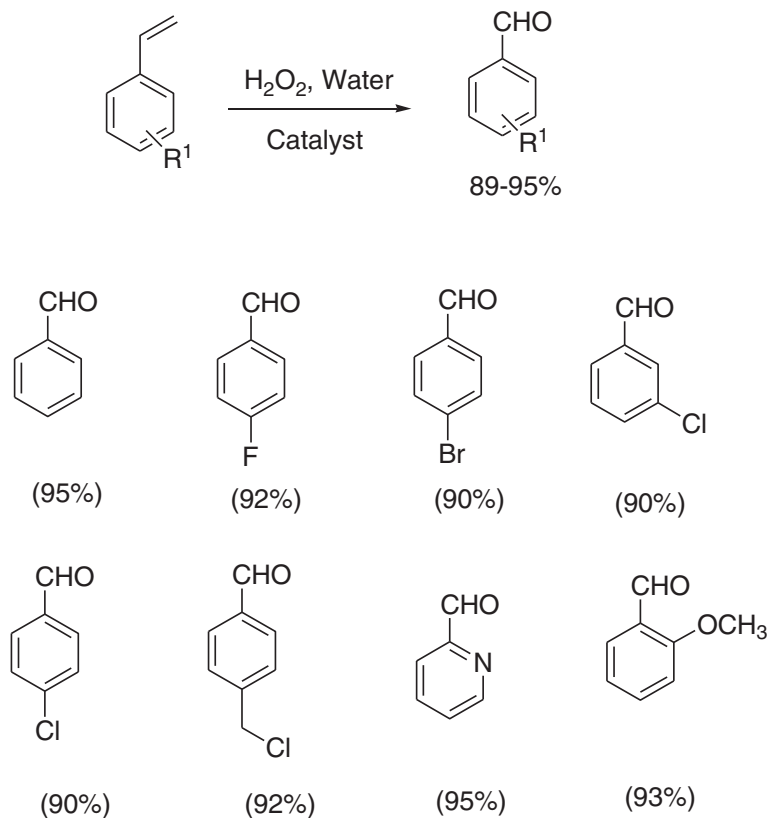
Scheme 5 Hydration of nitriles by nano-Fe@SiO₂Ru catalyst (reproduced by permission from Royal Society of Chemistry, reference 7).

In contrast, when dopamine is used as the anchoring ligand for Cu nanoparticles, the ensuing catalyst is active for C-S coupling and completely inactive for the Huisgen cycloaddition reactions [28].

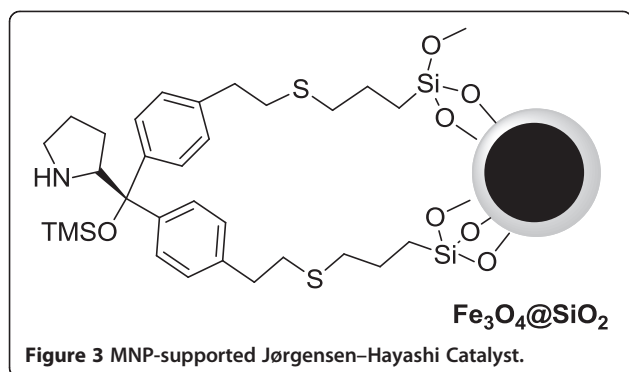
A conceptually simple strategy has been advanced for synthetic chemists wherein synthesis of Fe₃O₄@SiO₂Ru for the hydration of nitriles in aqueous media occurs

(Scheme 5) via sequential addition of reagents in one-pot to generate and to coat the magnetic particles [29].

Simple mesoporous silica supported iron oxide nanoparticles have been used for the oxidation of alkenes using hydrogen peroxide in water [30]. Styrenes and their substituted derivatives are converted to respective aldehydes in excellent yield (Scheme 6). It is important



Scheme 6 Mesoporous silica supported iron oxide nanoparticles for the oxidation of alkenes using hydrogen peroxide in aqueous medium.



to note that no other side products or oxidation of aldehydes are observed during the reaction. The optimized protocol is suitable to a range of electron-withdrawing and electron-donating substituents localized in all positions of the aromatic ring.

Supported iron oxide nanocatalysts have been the focus in various important catalytic applications because of their low cost and toxicity, ready availability, and environmentally benign nature [7,31].

Asymmetric synthesis using magnetic nanoparticles

The utility of MNP supported catalysts in asymmetric synthesis has been illustrated in an example where superparamagnetic nanoparticle-supported (*S*)-diphenylprolinol trimethylsilyl ether has been employed for the asymmetric Michael addition in water [32]; Jørgensen-Hayashi catalyst, ((*S*)-diphenylprolinol trimethylsilyl ether) on silica superparamagnetic nanoparticle support, (Figure 3), was prepared by a multistep synthetic procedure.

As-synthesized MNP-supported Jørgensen-Hayashi catalyst accomplished the asymmetric Michael addition of enolisable aldehydes to nitroalkenes in water; the corresponding products were obtained in moderate to good yields (up to 96%), and with good enantioselectivity (up to 90% ee) (Scheme 7).

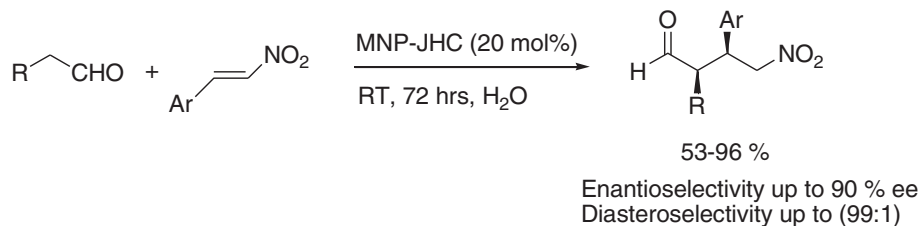
Significantly, the reaction was conducted between trans- β -nitrostyrene and propanal in water to deliver high yield of the product (85%). A variety of nitroalkenes and aldehydes performed well under these optimized reactions

conditions and the corresponding products were obtained in good to excellent yields in benign aqueous medium.

Synthetic processes using alternative energy input in combination with nano-catalysts thus shorten the reaction time and that eliminate or minimize side product formation [8]. This concept when used in the syntheses of pharmaceuticals, fine chemicals, and polymers may pave the way towards the greener and more sustainable approach to chemical syntheses. Newer developments on these themes, especially involving benign reaction media such as water and polyethylene glycol (PEG), in conjunction with photo activation [33], MW and ultrasonic irradiation, and/or ball-milling under solvent-free conditions, may help realize sustainable pathways for chemical synthesis and transformations, including the generation of novel nano-catalysts [7,8].

Nano-catalysts, especially those that can be recycled magnetically, are of special value in synthetic domain. The modifications of the surface of MNPs with other nanometals are of interest for catalysis. Such post-synthetic alterations using organic ligands [20], mimicking organocatalysis, allows the adsorption of catalytically active metal nanoparticles, which provide identical or better reactivity than the corresponding homogeneous catalysts [25,34]. The enhanced dispersity of the MNPs in common solvents is an added advantage, since it exposes the surface-bound active reaction sites for the reactants in ideal fashion. In foreseeable future, the design of novel magnetically retrievable heterogeneous asymmetric catalysts supported on Fe_3O_4 nanoparticle systems will find significant applications in asymmetric hydrogenations [35], asymmetric C-C bond formation reactions, and in asymmetric cycloaddition reactions, especially under continuous-flow conditions in microreactors [36].

Bare iron oxide nanoparticles provide ready access to oxidation and oxidative coupling reactions, while the reduced $\text{Fe}(0)$ NPs facilitate dehydrogenation, hydrogenation, couplings and reductive processes. The incorporation of a second metal serves to further expand the catalytic abilities of Fe and incorporation of oxides on to silica has been utilized for the biodiesel production [37]. Supporting various ligands on the magnetic nanoparticles facilitates synthesis of several chemical entities with sustainable advantages, predominant being the magnetic separation



and recyclability aspects [38,39]. Incorporation of copper provides an easy preparation of propargylamines via a multicomponent reaction [40] and asymmetric nanocatalysis is facilitated by support of heterocyclic carbenes as chiral modifiers [41].

Magnetic nanoparticles are garnering special attention in the emerging area of flow chemistry. The dual function of confinement and agitation of the nanoparticle-bound catalyst can be achieved in a reactor by means of a rotating magnetic field, which avoids the potential problems of clogging membranes or filters that are eminent barriers for immobilized catalysts. Reiser et al. have demonstrated this successfully in a close circuit reactor for the asymmetric benzoylation of racemic 1,2-diols using a copper (II)-azabis(oxazoline) catalyst, which had been covalently attached to carbon-coated cobalt nanoparticles [42]; magnetic field induced flow mixing is beginning to make critical impact for handling of slurries and precipitates in modern small footprint flow reactors [43].

Competing interests

The author declares that he has no competing interest.

Authors' information

Prof. Rajender S. Varma was born in India (Ph.D., Delhi University 1976). After postdoctoral research at Robert Robinson Laboratories, Liverpool, UK, he was faculty member at Baylor College of Medicine and Sam Houston State University prior to joining Sustainable Technology Division at US Environmental Protection Agency in 1999. He has over 40 years of research experience in management of multi-disciplinary technical programs ranging from natural products chemistry to therapeutics and development of environmentally friendlier alternatives for synthetic methods using microwaves, and ultrasound etc. More recently, he is focused on greener approaches to assembly of nanomaterials and sustainable applications of magnetically retrievable nano-catalysts in benign media. He is member of the editorial advisory board of several international journals and published over 390 scientific papers and has been awarded 12 US Patents.

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