SYNTHESIS OF METAL/METAL OXIDE NANOMATERIALS FOR ORGANIC TRANSFORMATIONS: A REVIEW

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Abstract— Nanomaterials are a diverse class of materials with dimensions ranging from 1 to 100 nm, offering exceptional surface areas and unique properties in magnetic, electrical, optical, mechanical, and catalytic fields, which can be precisely controlled. Researchers are exploring green, sustainable, and economic techniques for organic transformations of raw materials, with nanostructured catalysts being preferred due to their surface-active sites, high recovery rate, and ease of synthesis. The various nanocatalyst including mixed metal oxides, magnetic, core-shell, polymer-based, graphene-based, nano-supported have been employed as nanocatalyst in organic transformations. Metal/metal oxides nanocomposites, in particular, have emerged as viable alternatives to conventional materials in various fields. These nanocatalysts offer advantages such as increased surface area, selectivity, and cost-effectiveness. They are also inexpensive, stable, and can be easily recycled and reused for multiple cycles. The current review outlines the various types of metal/metal oxides nanomaterials involved in catalysis for organic transformations.

Index Terms— metal/metal oxides, nanocatalyst, nanomaterials, organic transformations

I. INTRODUCTION

Nanotechnology is considered one of the important technologies of day-to-day developments in research nbecause to its exceptional mechanical, electromagnetic, and optical characteristics. Nanomaterials are man-made, possessing special properties and functions with, at least one external dimension that measure 100 nanometres [1-4].

These nanomaterials include nano-objects such as nanoparticles, nanofibers (rods, tubes) and nanoplates, which can consist of different materials in the form of alloy and intermetallic compound and having different structures like crown jewel, hollow, core-shell and alloy structure. Metal/metal oxides nanomaterials are synthesized by physical, chemical and biological method, it involves Thermal and photochemical deposition, chemical vapour deposition, sputtering, sol-gel, co-precipitation, micro-emulsion, hydrothermal, solvothermal etc (Fig.1). The increasing uses of such synthetic nanomaterials have increased the scope of its application in different fields includes environmental, energy harnessing, biomedical sector and catalysis [2-4].

Fig.1- Different techniques of synthesis of Nanomaterials

Nanocatalysis: The substance (size in 1-100 nm) use as catalyst which alters the rate of chemical reaction is called as nanocatalyst. The use of catalyst in chemical technology is of great importance because use of small amount with high activities is preferable for economic and environmental conditions [5-6]. Two major classifications of catalysis based on the physical state of the catalyst in a chemical reaction are homogeneous catalysis and the heterogeneous catalysis. Both the types of catalysis possess their own advantages as well as disadvantages. Nanocatalyst is a linkage between homogeneous and heterogeneous catalyst because having excellent catalytic ability and selectivity (as homogeneous catalysts), easy recovery and reuse (as heterogeneous catalysts) [7].

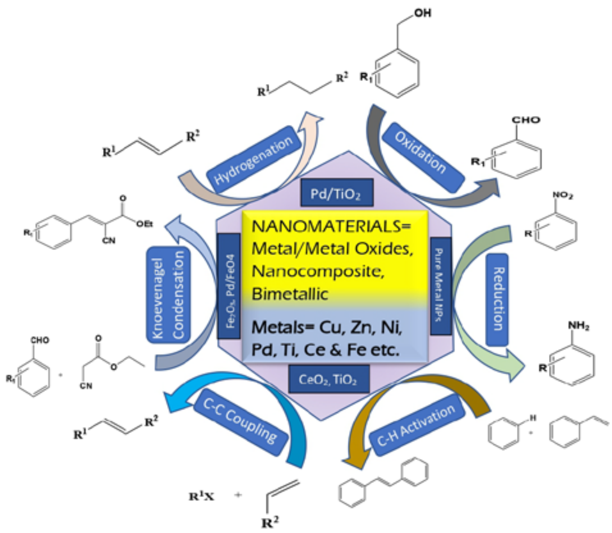
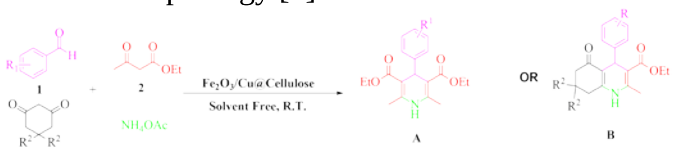


FIG.2- APPLICATIONS OF NANOMATERIALS AS CATALYST FOR ORGANIC TRANSFORMATIONS

In last decade various types of nanomaterials synthesized by researchers and investigate their catalytic activity on different organic transformation reactions. R. Chaudhury et al. synthesized CuO nanocatalyst using Lantana camara flower extract and examined their catalytic activity on aza-Michael reaction [8]. A. Muthuvinothini et al. prepared metal oxides and use as catalyst for reduction aldehyde reactions [9]. G. Rathee et al. fabricated gold supported NiAlTi nanocatalyst and use as catalyst for synthesis of Xanthene, 1,4-Dihydropyridine, Pyran derivatives [10]. Present review article covers the synthesis and characterization of numerous metal/metal oxides nanoparticles and nanocomposites. Studied catalytic behaviour of nanocatalyst on various organic transformations.

II. VARIOUS METAL/METAL OXIDES NANOMATERIALS FOR ORGANIC TRANSFORMATIONS

Ali Maleki, et al. synthesized an efficient magnetic γ-Fe_2O_3/Cu@cellulose bionanocomposite and effectively used it in the multicomponent condensation reactions for the synthesis of 1,4 dihydropyridine and polyhydroquinoline derivatives starting from simple and readily accessible precursors under solvent-free conditions at room temperature. FE SEM and TEM images of the bionanocomposite were indicated a narrow size of less than 30 nm and a distribution of Cu and γ-Fe_2O_3 nanoparticles distributed on the biomatrix with uniform morphology [1].



Scheme1: γ-Fe_2O_3/Cu@cellulose-catalyzed green synthesis of A and B

Mohd Umar Khan and Zeba N. Siddiqui et al. fabricated a highly recyclable catalyst Ce@STANPs/ZrO2 with an average particle size of 6 to 7 nm. The heterogenous Ce@STANPs/ZrO2 catalyst reported for the first time the synthesis of isatin-based imidazoles under microwave irradiation in water with a short reaction time [2].

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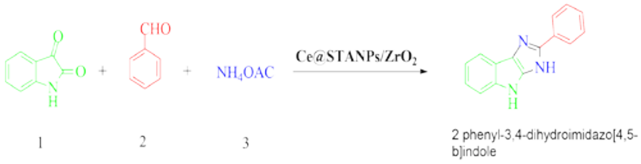
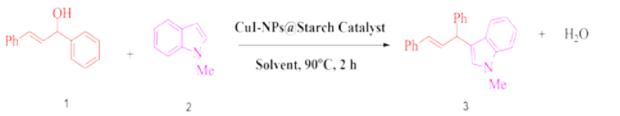


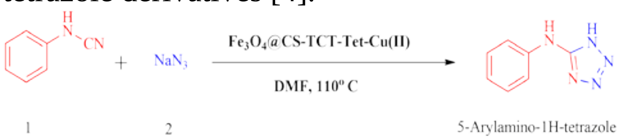
Scheme2: Synthesis of 2-phenyl-3,4-dihydroimidazo[4,5-b] indole

Sadhucharan Mallick et al. reported cuprous iodide nanoparticles (CuI-NPs@Starch) in aqueous medium and characterized by transmission electron microscopy, scanning electron microscopy, X-ray powder diffraction, energy-dispersive X-ray spectroscopy and atomic absorption spectra analysis. The newly synthesized CuI NPs on starch have been demonstrated first time as an efficient catalyst for the regioselective 3-allylation reaction of N-substituted indoles as well as ring-substituted indoles using various allyl alcohols under moisture and air insensitive conditions [3].



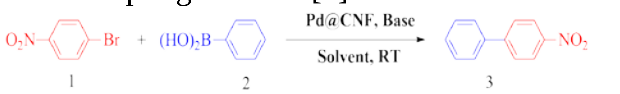
Scheme3: Regioselective 3-allylation reaction of ring- and N-substituted indoles

Mahmoud Nasrollahzadeh, et al. synthesized of magnetic chitosan functionalized tri chlorotriazine-5-amino- 1H-tetrazole copper (II) complex (Fe3O4@CS-TCT-Tet-Cu (II)). In given synthesis methodarylcyanamides and N-sulfonyl-N-arylcyanamides used for 5-aryl amino- 1H-tetrazole and N-sulfonyl-N-aryl tetrazole derivatives [4].



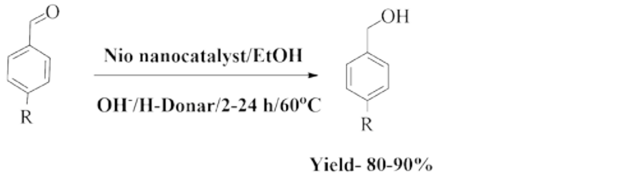
Scheme4: Synthesis of 5-arylamino-1H-tetrazole and N-sulfonyl- N-aryl tetrazole derivatives

Anindita Dewan, et al. fabricated cellulose-supported heterogeneous nanocatalyst Pd@CNF and applied it in the Suzuki-Miyaura cross-coupling reaction [5].



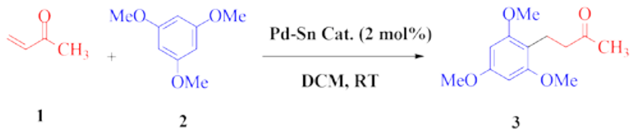
Scheme5: Optimization of reaction condition for Suzuki- Miyaura cross-coupling reaction.

Muthuvinothini and S. Stella reported the synthesis of Nio catalyst using aqueous immature fruit extract of Cocos nucifer through a green pathway. The catalytic activity of the synthesized nanoparticles was examined for the reduction of aromatic benzaldehydes [6].



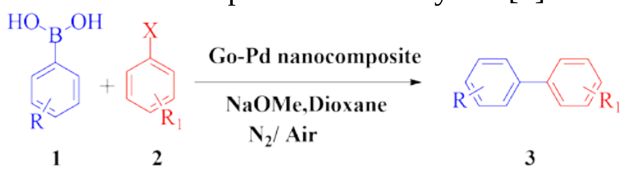
Schem6: Reduction of aromatic benzaldehydes.

Debjit Das, et al. Prepared the Pd -Sn heterobimetallic and effect of ligand and the coordination mode of enone with "Pd−Sn" heterobimetallic system were studied through Kinetic and DFT studies [7].



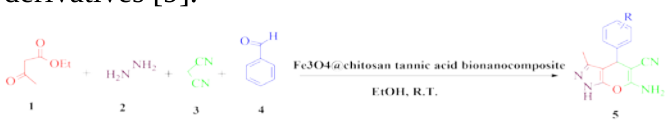
Scheme7: Synthesis of 1,4-oxathiophene core

K.S. Jithendra Kumara et al. developed a novel technique for the Graphene Oxide (GO) supported palladium nanocomposite (Pd NC) as a highly effective heterogeneous catalyst. The prepared GO-Pd NC acts as a catalyst precursor for the Suzuki coupling reaction. The catalyst is efficient under different reaction conditions, such as reaction temperature, time, solvent, and catalyst loading. The catalyst was useful for Suzuki reaction up to 5 reaction cycles [8].



Scheme8: Synthesis of GO-Pd NC for Suzuki coupling reaction

Maryam Kamalzare et al. fabricated Fe_3O_4@chitosan tannic acid bionanocomposite through in situ method by using chitosan and tannic acid as a natural source. This study represents an efficient practical method for the preparation of pyranopyrazole and its derivatives [9].



Scheme9: Synthesis of Fe_3O_4@chitosan-tannic acid bionanocomposite and its catalytic activity

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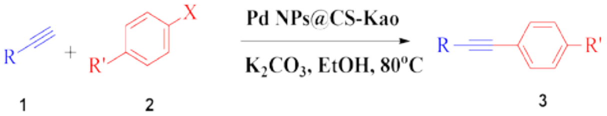
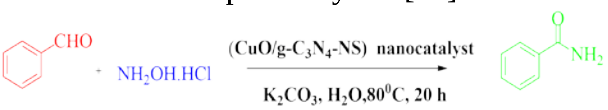
in the synthesis of pyranopyrazole and its derivatives

Mahmoud Nasrollahzadeh et al. construct Pd nanoparticles (NPs) supported on a novel Schiff base modified chitosan-kaolin (Pd NPs@CS-Kao) using natural resources and studies the Sonogashira coupling reaction (SCR) between aryl halides and acetylenes under aerobic condition [10].



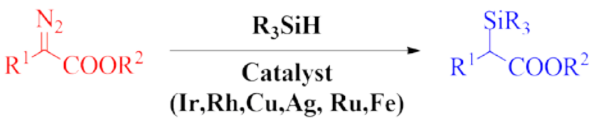
Scheme10: Pd NPs@CS-Kao catalyzed SCR of terminal alkynes with different aryl halides

Robabeh Mohammadi et al. describe a brilliant strategy to synthesize graphitic carbon nitride (g- C3N4) nanosheets decorated with copper oxide nanorods (CuO NRs). In the given synthesis, primary amides are prepared in water using CuO/g-C_3N_4-NS as a catalyst. The synergistic effect between the CuO effect and g-C_3N_4 nanosheets is the main factor in the formation yield. The reusability of CuO/g-C_3N_4-NS was verified through several reactions. This study will help carry out various developments for synthesizing primary amides in water. The morphology of CuO and its synergistic effect with g- C_3N_4 nanosheets play a vital role in the product yield [11].



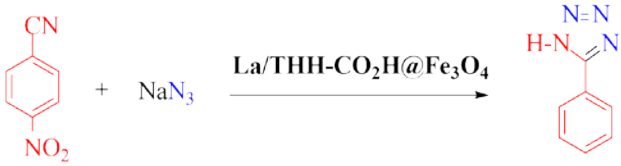
Scheme11: Synthesis of amide from aldehyde using CuO/g- C_3N_4 NS catalyst

Marios Kidonakis and Manolis Stratakis reported the catalysis of carbene insertion from electron-deficient compounds such as α-diazocarbonyl compounds into hydrosilanes by Au nanoparticles on TiO_2. For example, treatment of ethyl diazoacetate 1 with triethylsilane in the presence of 1 mol% Au/TiO_2 in DCE as the solvent affords α-silyl acetate 2 in good to excellent yields (Scheme 1) along with the reduction product (C=N_2 to CH_2). The reaction extends to a variety of diazoketone and silane substituents [12].



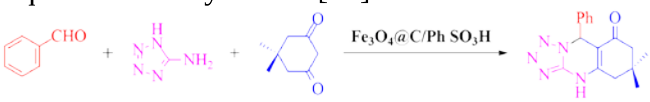
Scheme12: Conversion of α-Diazoesters to Hydrosilanes using Au nanoparticles on TiO_2

TaiebehTamoradi et al. developed a facile magnetic nanocatalyst using La on Fe3O4 nanoparticles pre-functionalized with tetrahydroharman-3-carboxylic acid ligand. The composite efficiently synthesizing 5-substituted 1H-tetrazoles, 1H-substituted 1H-tetrazoles, and tetrazolopyrimidine derivatives [13].



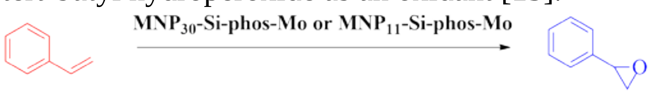
Scheme13: Fabirication of 5-substituted-1H-tetrazoles over La/THH-CO_2H@Fe_3O_4

Asadollah Hassankhani et al. Reported an eco-friendly and cost-effective Fe_3O_4@C/Ph SO_3H heterogeneous catalyst for direct synthesis of tetrazoloquinazolines. The method involved one-pot couplings of aromatic aldehydes, dimedone, and 1,3-cyclohexanedione ketones, resulting in high yields of various derivatives of tetrazoloquinazolines and avoiding dangerous liquid acids in synthesis [14].



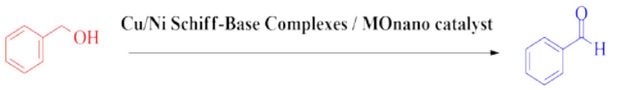
Scheme14: One Pot synthesis of tetrazoloquinazolines using Fe_3O_4@C/Ph SO_3H catalyst

Cristina I. Fernandes *et al.* synthesized Iron oxide magnetic nanoparticles (MNP_{30}-Si-phos-Mo, MNP_{11}-Si-phos-Mo, and MNP30 Sius-phos-Mo as catalysts) with different sizes (11 and 30 nm) and coated them with silica to allow the grafting of an organic phosphine ligand. The silica layer was prepared using the Stöber method, resulting in less aggregation and better coordination of the moiety. Structural characterization confirmed successful synthesis, and the nanomaterials were successfully used in olefin epoxidation using tert-butyl hydroperoxide as an oxidant [15].



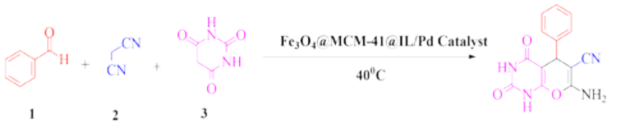
Scheme15: Catalytic epoxidation of styrene using MNP_{30}-Si-phos-Mo, MNP_{11}-Si-phos-Mo, and MNP30 Si_{us}-phos-Mo as catalysts

Sameerah I. Al-Saeedi *et al.* construct two Schiff-base ligands by condensation of 2-amino-3 hydroxypyridine with either 3-methoxysalicylaldehyde or 4-nitrobenzaldehyde. Then, using a sonochemical method, the nanosized Cu(II) and Ni(II) complexes, ahpvCu, ahpnbCu, and ahpvNi, were obtained. When the oxidation of Benzyl alcohol to benzaldehyde is performed in DMSO with H_2O_2 acting as the oxidizing agent, the prepared nanosized Schiff-base complexes and their MOs deliver exceptional catalytic performance. The complexes that have been prepared are suitable options for studying the catalytic conversions of alcohols and other organic substances [16].



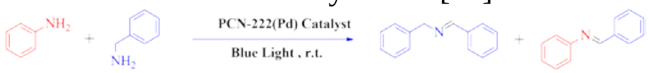
Scheme16: Catalytic oxidation of benzyl alcohol to benzaldehyde using Cu/Ni Schiff-Base and their metal oxide nanoparticle

Somayeh Abaeezadeh et al. developed an efficient nanocatalyst for the synthesis of biologically active pyrano[2,3-d] pyrimidines was prepared using a novel magnetic mesoporous silica (Fe3O4@MCM-41@IL/Pd) that contains palladium. Good incorporation/immobilization of both organic and inorganic moieties into/onto the catalyst framework, as well as high stability, were confirmed by the characterization techniques. Under solvent-free conditions, this nanocatalyst produced high to excellent yields of pyrano[2,3-d] pyrimidine products. With no appreciable loss in efficiency, the catalyst could be magnetically recovered and used at least eleven more times [17].



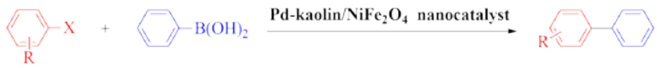
Scheme17: Preparation of biologically active pyrano[2,3-d] pyrimidines using Fe_3O_4@MCM41@IL/Pd

Aitor Bermejo-López et al. discovered a new synthetic process that produces palladium-metalated PCN-222 in just one hour. The type of metal center significantly impacts catalytic activity in photo-oxidative cross-condensation of imines. Under blue light irradiation, anilines and benzylamines react to give imines selectively, using PCN-222(Pd) as a catalyst. The study demonstrates the application of specific conditions to substrates like o-phenylenediamine, demonstrating isolation and transformations for various building blocks. PCN-222(Pd) exhibits good recyclability, maintaining yields over 90% after five runs. Scalability was tested in cross-condensation between aniline and benzylamine [18].



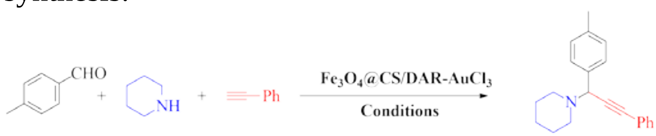
Scheme18: Fabrication of photo- oxidative cross-condensation of imines using palladium-metalated PCN-222 catalyst

Melike Çalıs ̧kan, and Talat Baran et al. construct an eco-friendly, inexpensive, and magnetically retrievable catalyst using palladium nanoparticles on kaolin/spinel nickel ferrite composite (Pd-kaolin/NiFe2O4). The Pd-kaolin/NiFe2O4 catalyst's structural and morphological properties were investigated, and its catalytic potential was tested in a Suzuki cross-coupling reaction. The design was found to be useful and stable for constructing biaryls [19].



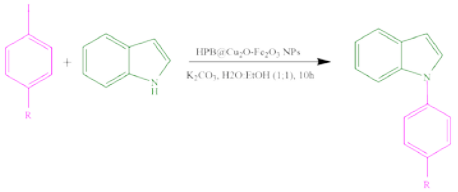
Scheme19: Suzuki cross-coupling reaction of biaryls using Pd-kaolin/$NiFe_2O_4$ nanocatalyst

Maryam Nourmohammadi et al. synthesized Magnetic DAR-chitosan by combining magnetic chitosan [Fe3O4@CS] with diacetylresorcinol as a cross-linking agent. The Schiff base precursor coordinated with Au (III) to form an Au (III) Schiff base complex (Fe_3O_4@CS/DAR-$AuCl_3$). The structure was studied using various techniques, and the synthesized Fe_3O_4@CS/DAR-$AuCl_3$ was used as a sustainable catalyst in pharmaceutical synthesis.



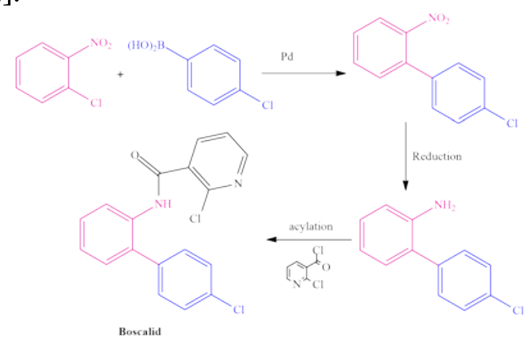
Scheme20: A^3 coupling reaction using Fe_3O_4@CS/ DAR-$AuCl_3$

G. Singh et al. prepared ultrafine hybrid Cu_2O–Fe_2O_3 NPs using hexaphenylbenzene derivative as nanoreactors and stabilizers. These NPs are an efficient and recyclable photocatalytic system for C–N coupling between aryl halides and amines, and exhibit high efficiency in synthesizing biologically important N-substituted carbazole derivatives [21].



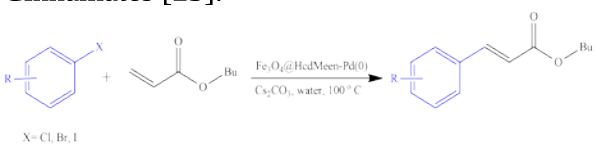
Scheme21: Synthesis of N-substituted carbazole derivatives

B. Takale et al. demonstrated the synthesis of Boscalid through Suzuki-Miyara coupling using Pd catalyst. They found high yield around 97% [22].



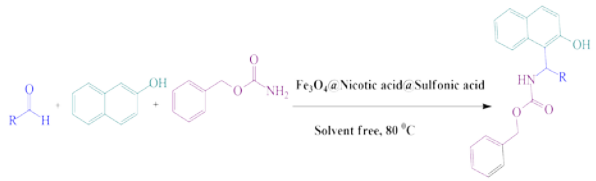
Scheme22: Synthesis of Boscalid using Pd catalyst

Muhammad Aqeel Ashraf *et al.* developed Fe_3O_4 @HcdMeen Pd(0) nanocatalyst for Heck C–C Cross Coupling Synthesis of Butyl Cinnamates. They observed that the novel catalyst is easily recoverable, efficient, and reusable and obtained high yield of Butyl Cinnamates [23].



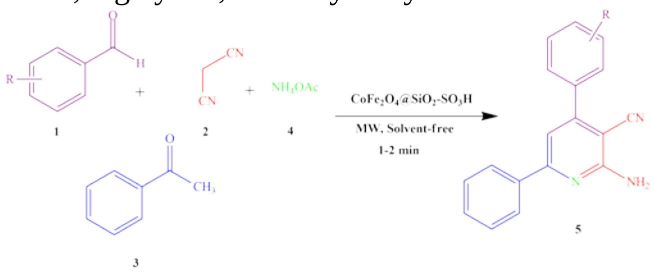
Scheme23: Synthesis Butyl Cinnamates through Heck C–C Cross Coupling

Ardeshir Khazaei *et al.* prepared a magnetic reusable catalyst, Fe_3O_4@nicotinic acid @sulfonic acid chloride, which was studied using various techniques. This catalyst was used for the one-pot synthesization of 1-carbamato-alkyl-2-naphthol derivatives in high yields under solvent-free conditions.



Scheme24: The synthesis of 1-carbamato-alkyl-2-naphthols by nano catalyst

Zahra Hosseinzadeh *et al.* fabricated modified $CoFe_2O_4$ magnetic nanoparticles with chlorosulfonic acid offers an efficient and simple method for synthesis and recovery of an organic-inorganic hybrid heterogeneous catalyst. The nanoparticles can be used for the preparation of 2-amino-4,6-diarylnicotinonitrile under microwave irradiation. The synthesis process offers advantages like shorter reaction times, high yield, and easy recrystallization.



Scheme25: Fabrication of 2-amino-4,6diarylnicotinonitrile in the presence of $CoFe_2O_4$@SiO2-SO3H

III. CONCLUSION

Nanomaterials, which have a long history and are generally considered nanomaterials with dimensions between 1-10 nm, have shown significant progress in various fields. They possess unique features such as high surface areas, magnetism, quantum effects, antimicrobial activity, and high thermal and electrical conductivities. Metal-based materials have shown high catalytic activities, and better dispersion can be achieved through dispersion on 2D sheets of other nanomaterials. The nanomaterials family includes carbon-based nanomaterials, nanoporous materials, core-shell materials, ultrathin 2-dimensional nanomaterials, and metal-based nanomaterials. Carbon-based nanomaterials, including fullerenes, carbon nanotubes, carbon-based quantum dots, graphene, and carbon nanohorns, have been extensively explored for various applications due to their high surface areas, rapid charge transfer properties, and high mechanical strength. In this review discussed these types of nanocatalyst for different organic reactions. The catalyst is recyclable and can be reused multiple times without loss of catalytic activity.

IV. ACKNOWLEDGMENT

The author recognizes and appreciates the significance of the contributions of previous and fellow researchers in the field of heterogenous nanocatalysis and their reactions.

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