Review Article

Status of Organometallics and Catalysis Research in India: N-Heterocyclic Carbene (NHC) Ligands for Organometallic Catalysis

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The core activities in the field of organometallic chemistry and catalysis research in India are summarized in this article. It aims to portray the depth and diversity of the chemistry led by different research groups across the country. Selected works on the chemistry of metal–NHC compounds are highlighted.

Keywords: Organometallics; Catalysis; N-heterocyclic Carbenes (NHC); Metal-catalyzed Transformations

Background

Organometallics is the chemistry of metal-carbon bonds. The widespread interest on organometallic compounds stems largely from their intriguing structure-bonding relationships and vast applications in organic transformations. Organometallic chemistry plays a prominent role for the synthesis of fine chemicals, natural products, pharmaceuticals, agricultural products polymers and other useful materials. In recent years, sustainable processes and products are developed utilizing new generation metal-based catalytic systems. The relevance of organometallic catalysis is evident in renewable energy research. The 2005 (Casey, 2005; Chauvin, 2006) and 2010 (Nagendrappa and Kumar, 2011; Croft, 2010) Nobel prizes in chemistry are the recognition of the immense and continued impact of organometallics in chemical synthesis. The aim of this article is to present a bird's-eye view of contemporary organometallic chemistry and catalysis research in India. A comprehensive coverage is impractical within the scope of this article. Rather, it highlights the core activities of different research groups, followed by a focused and short description of the chemistry of metal–N-heterocyclic carbene (M–NHC) compounds – a self-serving choice. Efforts are made to cover significant activities that fit within the broader

definition of organometallics. Any inadvertent omission, as it may well be, is regretted.

Metal Salts in Catalysis

There have been sustained activities on the direct use of metal salts as catalysts for a wide range of organic transformation reactions. Chandrasekaran group (IISc) explored the chemistry of MoS_4^{2-} and WSe_4^{2-} as versatile sulfur and selenium transfer reagents, respectively, to access functionalized disulfides, diselenides, selenoamides, selenoureas, and cyclicselenide derivatives (Sureshkumar *et al.*, 2012; Suresh *et al.*, 2005; Devarajulu *et al.*, 2010; Devarajulu *et al.*, 2007a; Devarajulu *et al.*, 2007b). At IITK, Iqbal group research focused on developing synthetic methodologies involving cobalt-catalyzed functional group transformations for applications in synthetic chemistry (Iqbal *et al.*, 1994; Bhatia *et al.*, 1993; Punniyamurthy *et al.*, 1993; Iqbal *et al.*, 1991). Periasamy group (UH) demonstrated carbonylation of organic compounds using metal carbonyls (Beesu and Periasamy, 2011; Beesu and Periasamy, 2012; Periasamy *et al.*, 2008). $\text{Fe}(\text{CO})_5$ was employed for the carbonylation of alkynes to obtain cyclobutenediones and anhydrides (Periasamy *et al.*, 2004). In addition, they developed new synthetic methodology involving $\text{TiCl}_4/\text{R}_3\text{N}$ reagent systems

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(Periasamy, 2002; Bharathi and Periasamy, 1999; Bharathi and Periasamy, 2000; Periasamy *et al.*, 2010; Karunakar and Periasamy, 2006). Kotha group (IITB) exploited various ruthenium catalyzed metathesis reactions as key steps to access ring annulated products (Kotha and Gunta, 2017; Kotha *et al.*, 2015; Kotha *et al.*, 2011; Kotha and Chavan, 2010; Kotha and Khedkhar, 2009; Kotha *et al.*, 2009). Further, they exploited Suzuki–Miyaura cross-coupling strategy to access diverse polycyclic aromatics (Kotha and Ghosh, 2004; Kotha and Mandal, 2006; Kotha and Mandal, 2009; Kotha *et al.*, 2007; Kotha *et al.*, 2002). M. Lakshmi Kantam from IICT has exploited metal nanoparticles on different solid supports for a wide variety of organic transformations (Layek *et al.*, 2012; Chakravarti *et al.*, 2011; Kantam *et al.*, 2013; Arundhathi *et al.*, 2013; Kantam *et al.*, 2009). Ranu group (IACS) used metal nanoparticles as catalysts for a variety of organic transformation reactions, in particular, Pd and Fe nanoparticles for direct C–H functionalization (Saha *et al.*, 2010; Panja *et al.*, 2017; Chatterjee *et al.*, 2014; Kundu, *et al.*, 2013) and reduction reactions (Panja *et al.*, 2017; Dey *et al.*, 2012; Saha and Ranu, 2008).

Organometallic Chemistry

These activities had a thick organic chemistry flavor. Other research groups have pursued classical organometallic chemistry research that includes the synthesis of organometallic compounds, their reactivity studies and potential use in catalysis. S S Krishnamurthy (IISc) group designed and developed an array of P–N and P–N–P based ligands. Palladium-allyl, ruthenium-hydride and carbonyl cluster compounds supported by those ligands were synthesized and exploited in catalytic coupling and transfer hydrogenation reactions (Reddy *et al.*, 1992; Maji *et al.*, 2011; Mandal *et al.*, 2003; Venkatakrishnan, *et al.*, 2006; Venkatakrishnan, *et al.*, 2007; Mandal *et al.*, 2006). Chandrasekhar group (IITK) studied the organometallic chemistry of main group elements (Chandrasekhar *et al.*, 2009a; Chandrasekhar *et al.*, 2009b). Significant advances were made in the chemistry of organostannoxane and organotelluroxane compounds. Carboxylates, phosphate and sulphonates supported organotin molecular assemblies were synthesized and structurally characterized (Chandrasekhar *et al.*, 2007; Chandrasekhar *et al.*, 2005). Organostann-

oxane supported palladium nanoparticles were employed for the efficient cross-coupling reactions (Heck and Suzuki) (Chandrasekhar *et al.*, 2009; Chandrasekhar *et al.*, 2011). Bhaduri at Reliance Industries studied platinum and ruthenium carbonyl cluster complexes and exploited inorganic-oxides supported metal carbonyl catalysts for hydrogenation reactions (Bhaduri *et al.*, 1998; Bhaduri *et al.*, 1983; Basu *et al.*, 1986; Bhaduri *et al.*, 2001; Paul *et al.*, 2003). They also developed technologies for the industrial scale manufacture of polypropylene utilizing supported or modified Ziegler–Natta type of catalysts (Gnanakumar *et al.*, 2011; Bhaduri *et al.*, 2006; Thushara *et al.*, 2012). Singh group (IITB) works on the organoselenium and organotellurium compounds (Manjare *et al.*, 2012; Kumar *et al.*, 1990; Panda *et al.*, 1990). The coordination behavior of these compounds with Pd(II) has been studied (Chakraborty *et al.*, 2011a; Chakraborty *et al.*, 2011b) and the catalytic utility of these complexes are evaluated for cross-coupling reactions (Ghavale *et al.*, 2015; Chakraborty *et al.*, 2011a). Samuelson (IISc) group examines anti-cancer activities of half-sandwich metal complexes with heterocyclic ancillary ligands (Mitra *et al.*, 2012; Mitra *et al.*, 2014). Besides metal-catalyzed transformation reactions, (Dinda *et al.*, 2010; Kumar *et al.*, 2011) his group undertakes electronic structure calculations to gain insight on bonding and energetics of organometallic compounds (Bera *et al.*, 1999; Dindar *et al.*, 2012). E D Jemmis (UH/IISc/IISER-TVM) (Jemmis *et al.*, 2002; Jemmis *et al.*, 2003; Kiran *et al.*, 2002; Roy *et al.*, 2014; Mondal *et al.*, 2018) and R B Sunoj (IITB) (Jindal *et al.*, 2014; Reddi *et al.*, 2019; Kumarasamy *et al.*, 2017; Bhaskararao and Sunoj, 2015) offer theoretical perspectives on the structures, bonding and reactivities of organometallic compounds. Mathur (IITB) explored the cluster chemistry involving chalcogen-bridged mixed-metal carbonyl clusters (Datta *et al.*, 1994; Mathur *et al.*, 1994; Mathur *et al.*, 2000; Mathur *et al.*, 2000; Mathur *et al.*, 2002; Klose *et al.*, 2003) and their acetylenic derivatives (Mathur *et al.*, 1995; Mathur *et al.*, 1993; Mathur *et al.*, 1996; Mathur *et al.*, 1995). Gupta group (IITK) examined the structure-property relationship of organo-cobalt and vitamin B12 model compounds (Gupta *et al.*, 2011; Dutta *et al.*, 2011; Bhuyan *et al.*, 2008; Mandal and Gupta, 2007). The reactivity of the Co–C bond was examined by the reactions of organocobaloximes with

molecular oxygen and Br₂ (Kumar and Gupta, 2011; Mandal *et al.*, 2007; Kumar *et al.*, 2011; Dutta *et al.*, 2012). Elias (IITD) has developed cobalt and iron sandwich compounds bearing tetraphenylcyclobutadiene and cyclopentadiene derivatives (Kumar *et al.*, 2016). Several donor groups are attached on the Cp ring and their metal complexes are synthesized (Hazra *et al.*, 2017; Singh *et al.*, 2016). Palladacycles constructed by chemical modification of the Cp ring were utilized for organic transformation reactions (Singh *et al.*, 2015; Singh and Elias, 2011; Singh *et al.*, 2014). His group earlier pursued organometallic reactions on moieties derived from cyclophosphazene cores (Elias *et al.*, 2011; Kumar *et al.*, 2014; Kumar *et al.*, 2008; Keshav *et al.*, 2010; Kumar *et al.*, 2011). Lahiri (IITB) has employed well known Ru based catalysts for alcohol dehydrogenation, amide synthesis from primary and secondary amines and epoxidation reactions (Ray *et al.*, 2018; Ray *et al.*, 2014; Chowdhury *et al.*, 2011). Iron salts are also used for oxidation and nitrosation of terminal alkenes (Chowdhury *et al.*, 2012a; Chowdhury *et al.*, 2012b; Ray *et al.*, 2013). Jagirdar group (IISc) is engaged in examining heterocyclic X-H bond activation at highly electrophilic metal center (Nagaraja *et al.*, 2007; Mala *et al.*, 2017; Mala *et al.*, 2018; Ramaraj *et al.*, 2016). Their work has provided valuable insight on the H-atom exchange process between the η^2 -H₂ and hydride ligands on the metal. His group has also developed a protocol for the synthesis of metal and bimetal alloy nanoparticles employing amine-boranes with appropriate metal salts. The use of amine-borane serves dual purposes – to reduce the metal ions to its elemental form and the generated BNH_x polymer acts as stabilizing agent. Dissolution of the capping polymer in water afforded mesoporous metal nanosponges which are conveniently used for a variety of reduction reactions under mild conditions (Kalidindi *et al.*, 2011; Sanyal and Jagirdar, 2012; Kalidindi and Jagirdar, 2012; Ghosh and Jagirdar, 2018). Roy group (IICT/IITKGP/IIT Bhubaneswar) developed heterobimetallic chemistry for catalytic organic transformations (Das *et al.*, 2015; Roy and Roy, 2010; Das *et al.*, 2012). Notably, transition metal–tin (TM–Sn) systems are devised for allylation, propargylation, allenylation, selenylation, benzylation reactions (Choudhury *et al.*, 2005; Podder *et al.*, 2007a; Podder *et al.*, 2007b; Kundu and Roy, 2007). A. K. Singh group (IITD) has exploited Pd,

Ru, Ir compounds with organochalcogen ligands for cross-coupling, oxidation and transfer hydrogenation reactions (Bhaskar *et al.*, 2018; Prakash *et al.*, 2013; Dubey *et al.*, 2018; Saleem *et al.*, 2013). Pandey at BHU uses half-sandwich Ru arene complexes for a variety of purposes (Kumar *et al.*, 2014; Singh *et al.*, 2014). Singh expands the similar chemistry at IITI (Dwivedi *et al.*, 2018; Awasthi *et al.*, 2018). Ghosh at IITM has combined organometallic fragments with borane constructs (De *et al.*, 2018; Anju *et al.*, 2014; Saha *et al.*, 2017). Anantharaman (IITK) studies metal-NHC compounds and their catalytic activity towards cross-coupling reactions (Karthik *et al.*, 2014; Bhat *et al.*, 2019; Karthik *et al.*, 2013).

Organometallic Catalysis

Rao (IITK) explores palladium catalyzed cross-coupling reactions for organic synthesis. Stable triarylbismuths are effectively used as aryl transfer reagents under palladium-catalyzed conditions for a wide variety of C-C bond forming reactions (Shimada and Rao, 2012; Rao *et al.*, 2010a; Rao *et al.*, 2010b; Rao *et al.*, 2008). Sekar (IITM) group research emphasizes on metal nanocatalysts (Savitha *et al.*, 2016a; Savitha *et al.*, 2016b; Surya *et al.*, 2016; Ganapathy *et al.*, 2014; Sharma *et al.*, 2017). Further, they have studied metal-catalyzed reduction of α -keto amides and their functionalization (Mamillapalli and Sekar, 2015a; Mamillapalli and Sekar, 2014; Mamillapalli and Sekar, 2015b; Kotha *et al.*, 2014; Muthukumar and Sekar, 2017; Muthukumar *et al.*, 2018). Punniyamurthy (IITG) focuses on the transition-metal catalyzed stereoselective carbon-carbon and carbon-heteroatom bond formation (Talukdar *et al.*, 2019; Banerjee *et al.*, 2019; Satheesh *et al.*, 2018; Mahesh *et al.*, 2017; Mahesh *et al.*, 2016; Sengoden *et al.*, 2017; Sengoden and Punniyamurthy, 2013). Hotha (IISER Pune) group employs gold catalysts for the synthesis of glycosides and furanosides respectively (Hotha and Kashyap, 2006; Kayastha and Hotha, 2012; Thadke *et al.*, 2013; Vidadala *et al.*, 2011; Sureshkumar and Hotha, 2008). The metal-catalyzed C-H bond activation and functionalization is an important area of research in chemical synthesis. Several groups are independently leading this activity- notable names are Maiti (Achar *et al.*, 2018; Maji *et al.*, 2018; Deb *et al.*, 2017), Jeganmohan (Ramesh and Jeganmohan, 2017; Reddy and Jeganmohan, 2017; Manikandan *et al.*, 2016),

Anbarasan (Reddy *et al.*, 2017; Yadagiri *et al.*, 2016; Yadagiri and Anbarasan, 2015), Sundararaju (Kalsi *et al.*, 2018; Barsu *et al.*, 2017; Sen *et al.*, 2016), and Baidya (Mandal *et al.*, 2017; Dana *et al.*, 2018a; Dana *et al.*, 2018b).

Chattopadhyay group (CBMR, Lucknow) engineered new catalytic systems integrating noncovalent interactions on transition metal complexes to generate novel concepts and hypotheses in C–H activation chemistry. Moreover, they have developed series of new denitrogenative annulation strategies employing organometallic complexes for the construction of diverse nitrogen heterocycles (Bisht *et al.*, 2018; Smith III *et al.*, 2018; Bisht and Chattopadhyay, 2016; Hoque *et al.*, 2017). Gunanathan (NISER) group studies ruthenium catalyzed deuteration of organic compounds using D₂O to labelled products (Chatterjee *et al.*, 2016; Krishnakumar and Gunanathan, 2018; Chatterjee and Gunanathan, 2016; Chatterjee and Gunanathan, 2015; Thiagarajan and Gunanathan, 2019). Balaraman group (IISER-Tirupati) efforts lie in the domain of catalytic hydrogenation and dehydrogenation reactions (Midya *et al.*, 2015; Sahoo *et al.*, 2017; Sahoo *et al.*, 2018; Jaiswal *et al.*, 2017; Nandakumar *et al.*, 2015). Chikkali group (CSIR-NCL) uses metal catalysts bearing phosphorus ligands featuring supramolecular binding property for hydrophosphination, hydrogenation, hydroformylation, polymerization reactions (Koshti *et al.*, 2014; Koshti *et al.*, 2015; Pandey and Chikkali, 2015; Pandey *et al.*, 2018; Mote *et al.*, 2017). Biplab group (IISERK) is developing new synthetic methodology using manganese catalyst to construct new C–C bonds by alcohols activation (Barman *et al.*, 2018a; Maji and Barman, 2017; Barman *et al.*, 2018b; Jana *et al.*, 2018). Kundu (IITK) group has exploited alcohol as an alkylating agent to construct new C–C and C–N bonds for organic synthesis (Shee *et al.*, 2018; Paul *et al.*, 2018; Maji *et al.*, 2018; Chakrabarti *et al.*, 2017).

Heterogeneous Catalysis

Although most activities in organometallic catalysis are confined under homogenous conditions, there is a vibrant community who work on heterogeneous catalysis. Prof. B M Choudary from ICT, Hyderabad is the pioneer utilizing layered double-hydroxides, copper apatite, nanocrystalline MgO, OsO₄

incorporated in resins, for a range of reactions such as C–H activation, N-arylation of heterocycles, Claisen-Schmidt condensation, asymmetric epoxidation and dihydroxylation of olefin (Choudary *et al.*, 2005; Choudary *et al.*, 2004; Choudary *et al.*, 2002; Choudary *et al.*, 2001). P. Selvam from IITM has synthesized several heterogeneous catalysts based on mesoporous silica (MCM-41) and their aluminate derivatives. These catalysts are evaluated for selective oxidation, hydrogenation, wet air oxidation of aniline, reductive cleavage of azo compounds (Selvam *et al.*, 2011; Mohapatra *et al.*, 2002; Gomes *et al.*, 2005; Sonavane *et al.*, 2003). S B Umbarkar from NCL, Pune exploited *in situ* generated molybdenum oxo-peroxo complexes as recyclable catalysts for oxidation reactions (Biradar *et al.*, 2008; Chandgude *et al.*, 2012; Nguyen *et al.*, 2012; Chandra *et al.*, 2013). In recent time, V. Polshettiwar group from TIFR, Mumbai developed dendritic fibrous nano-silica (DFNS) based nanocatalysts, such as TaH supported on silica, single atoms and nanoparticles of metal supported on amine and polyamine functionalized DFNS, for various range of catalytic transformations and CO₂ capture-conversions (Bayal *et al.*, 2017; Polshettiwar *et al.*, 2011; Polshettiwar *et al.*, 2009; Polshettiwar *et al.*, 2010; Singh and Polshettiwar, 2009).

Books on Organometallic Chemistry

The continued tradition of organometallic chemistry is reflected in the numbers of books written by Indian authors. Prof. R C Mehrotra offered an introductory account of organometallic chemistry (Mehrotra, 2000). V Chandrasekhar has authored a book on Inorganic and Organometallic polymers (Chandrasekhar, 2005). Anil Elias and B D Gupta (Gupta and Elias, 2010; 2013) have written a modern book on organometallics that has become an essential handbook for students and teachers as well. Anil Elias has recently introduced a well-researched book on the chemistry of the p-block elements (Elias, 2018). S. Bhaduri and D. Mukesh have written a book on homogeneous catalysis with particular emphasis on industrial applications (Bhaduri and Mukesh, 2014).

Metal–N-heterocyclic Carbene Complexes

Arduengo reported the first stable NHC (1,3-diadamantylimidazole-2-ylidene) in 1991 (Scheme 1). Initially NHCs were viewed as neutral 2-e donor

Scheme 1: Line drawing of a NHC

alternatives to phosphines and the synthesis to metal-NHC compounds was considered difficult than their phosphine counterparts. However, NHCs soon found numerous applications, and prominently in organometallic catalysis. Several synthetic protocols are developed over the years to access metal-NHC compounds. It is now well recognized that NHCs offer several advantages over phosphines. The NHC features a directional sp^2 -type lone pair compared to non-directional σ -type orbital for phosphine. The extent of π -back-bonding is lower in metal-NHC than in metal-phosphine complexes. These factors contribute to the stability of the metal-NHC compounds imposing higher electron density on the metal center. It has direct implications on their performances in catalytic processes. The Pd-NHC catalysts are in general more active and offer better turnover values for cross-coupling reactions than related phosphine-based catalysts. The evolution of Grubbs olefin metathesis catalysts is another example of the superiority of NHC ligands over phosphines.

The phosphine ligands played an important role in the early development of organometallic chemistry and catalysis. The footprint of Indian chemists in that era had been rather limited. In recent times, metal-catalyzed reactions involving metal-NHC compounds are the major topics of catalysis research and Indian chemists are making a sizable contribution to this field. We focus on research from selected groups engaged in exploring the chemistry of metal-NHC compounds.

Selected Works on the M-NHC Chemistry in India

Sarkar group at NCL Pune worked on the chemistry of metal-carbene complexes. A variety of reactions involving Fischer carbene complexes were studied including alkylation (Amin and Sarkar, 1995) and annulation, (Mathur *et al.*, 1998) regioselective 1,3-dipolar addition of organic azides (Chakraborty *et al.*,

2010) and rearrangement to ketone (Nandi *et al.*, 1992). Thermal rearrangement of aryl((arylmethyl)oxy)-carbene complexes of Cr and W afforded the corresponding ketones (Scheme 2). The

Scheme 2: Rearrangement of Fischer carbene complexes to ketones

rearrangement is general and applicable for aryl(allyloxy)carbene complexes as well. It was demonstrated that electronic tuning on the carbene carbon dictates the reactivity of the complexes. (Sathe *et al.*, 1996)

Sarkar exploited suitably modified Fischer carbene complexes for the reduction of Au salts and stabilization of nanoparticles (Samanta *et al.*, 2006). Hydrophilic Fischer carbene complexes are used as organometallic bio-markers (Samanta *et al.*, 2005). A ferrocene-analog of Grubbs catalyst was developed which show efficient RCM reactions (Scheme 3) (Maishal and Sarkar, 2002).

Scheme 3: An air-stable, reusable, ferrocene-analog of Grubbs catalyst

Ghosh at IITB initiated the chemistry of NHC based metal complexes, (Ramasamy and Ghosh, 2016; John and Ghosh 2010) focusing on exploring the potential of these compounds in biomedical applications, with a particular emphasis on developing Pd-NHC complexes for anticancer studies (Scheme 4a), (Kumar *et al.*, 2017; Ray *et al.*, 2007a; Kumar *et al.*, 2018) and also in catalytic applications. On the homogeneous catalysis front, Ghosh's group have demonstrated the utility of these complexes for simple Pd-mediated C-C cross-coupling reactions like the Suzuki, (Ray *et al.*, 2007b) Sonogashira (Scheme 4b)

(John *et al.*, 2009) and the Hiyama couplings (Modak *et al.*, 2015). Ghosh's group has successfully developed the more challenging bifunctional catalysis on NHC ligand platforms using nickel complexes for the chiral (Rao *et al.*, 2015) and achiral (Samantaray *et al.*, 2009; Ray *et al.*, 2009) variants of the base-free Michael addition reactions (Scheme 4c). Further, his group also reported Fe-NHC complexes for asymmetric Michael addition reactions (Prakasham *et al.*, 2018). They pursued applications of these complexes in industrially important and environmentally relevant atom-economic reactions like the hydroamination of activated olefins (Scheme 4d) (Kumar *et al.*, 2017; Dash *et al.*, 2010a) and the hydroamination of alkynes (Scheme 4e) (Katari *et al.*, 2012; Dash *et al.*, 2010b). Ghosh successfully performed silylation reactions in the form of hydrosilylation of carbonyl compounds using iron complexes (Scheme 4f) (Kumar *et al.*, 2014) and reported the catalytic utility of inert coinage metals like silver and gold in polymerization reactions for producing environmentally benign polylactide polymers using Ring Opening Polymerization of L-lactides (Scheme 4g) (Samantaray *et al.*, 2006; Ray *et al.*,

2006). In the domain of asymmetric catalysis, Ghosh's group has reported rhodium and iridium based precatalysts for the transfer hydrogenation of ketones (Scheme 4h) (Ramasamy *et al.*, 2017). Overall, Ghosh's group work represents the expansion of the application space, both biomedical and catalytic, of NHC based metal complexes using variation in carbene ligands and their well-defined metal complexes along with obtaining detailed mechanistic insights for further improvements of the property of interest.

Mandal at IISER Kolkata developed the chemistry of *mesoionic carbene* (MIC) which is also known as abnormal N-heterocyclic carbene (*a*NHC) where C2-position of imidazole ring is blocked with a phenyl group. By virtue of its strong nucleophilic nature (Thakur *et al.*, 2016; Sau *et al.*, 2016; Sau *et al.*, 2019; Hota *et al.*, 2018), a variety of transition metal complexes were prepared (Scheme 5). For example, Mandal and co-workers prepared halobridged C–H activated palladium dimers (Scheme 5(a)) (Hota *et al.*, 2015; Hota *et al.*, 2017), which are active catalysts for the Suzuki–Miyaura cross-coupling of a number

Ghosh's Work

Scheme 4: Catalysts developed in Ghosh laboratory and catalytic activities

of aryl chlorides at room temperature with low catalyst loading (up to 0.005 mol%) (Sau *et al.*, 2012; Sreejyothi *et al.*, 2018; Ahmed *et al.*, 2017). Importantly, complex (**a**) remained active for 10 successive catalytic runs without any loss of its activity, confirming the robustness of the Pd-*a*NHC bond. More recently, complex (**a**) was reported as an active catalyst for dehydrogenative cross-coupling using a variety of heteroarenes such as benzothiazole, benzoxazole, 2-formyl thiophene, furfural and N-methyl benzimidazole. Cu(I)-MIC complex (Scheme **5b**) was applied for cycloaddition of azides and alkynes at very low catalyst loadings and a wide range of triazoles in good to excellent yields were obtained (Scheme **5**) (Sau *et al.*, 2013). The MIC based iron (0) catalyst (Scheme **5c**) was used for selective hydrosilylation of various aldimines and ketimines for the synthesis of corresponding amines (Bhunia *et al.*, 2016). An aryl C-H activated nickel(II) cyclooctenyl complex (Scheme **5d**) was also isolated (Vijaykumar *et al.*, 2017). Complex (**5d**) catalyzes hydrohetero-arylation of various olefins through C-H bond activation, offering a highly atom-economical method for the synthesis of 1,1- or 1,2-diarylalkanes. Besides transition metal chemistry, Mandal and co-workers also prepared main-group metal complexes (**5e**), (**5f**)

and (**5g**) (Scheme **5**). (MIC)K(I) complex was utilized for the polymerization of *rac*-lactide in toluene at room temperature showing 96% conversion within 2.5 h (Bhunia *et al.*, 2017). (MIC)Al(III) complex (Scheme **5f**) and (MIC)Zn(II) complex (Scheme **5g**) also act as catalysts for the ring opening polymerization of various cyclic esters (Sen *et al.*, 2013).

Choudhury group (IISERB) has revealed an unprecedented chemistry of Rh-NHC backbone for the synthesis of highly conjugated, cationic and annulated organic molecules (Ghorai and Choudhury, 2014; Ghorai and Choudhury, 2015; Thenarukandiyil and Choudhury, 2015; Ghorai *et al.*, 2016; Thenarukandiyil *et al.*, 2016a; Thenarukandiyil *et al.*, 2016b; Dutta *et al.*, 2019). The rhodium(III) catalyzed C-H bond activation of aromatic, heteroaromatic and vinylic motifs was achieved by utilizing a novel conjugative action of NHC ligand as directing group followed by insertion of internal alkynes and subsequent annulation of various imidazolium salts (Scheme **6a-c**). They also reported a series of iridium(III)-NHC complexes with varying ring size for hydride-transfer reaction (Semwal *et al.*, 2014; Semwal *et al.*, 2017). Based on the bite angle and yaw angle of these complexes, their electronic properties were studied for the efficient hydride donor

Mandal's work

Transition-metal catalysts of Mesoionic Carbene (MIC)

Main-group-metal catalysts of Mesoionic Carbene (MIC)

Scheme 5: Catalysts developed in Mandal laboratory

ability to unsaturated organic molecules (Scheme 6d). Along this line, recently they developed α NHC-based iridium catalyst featuring a hybrid anionic amido-type supporting ligand for CO₂-conversion chemistry (Scheme 6e) (Semwal *et al.*, 2018; Kumar *et al.*, 2019). Further, they have introduced smartly designed benzimidazole-pyridinylidene-based NHC hybrid ligand constructed Ir and Ru complexes as a new class of ‘molecular switch’ to demonstrate stimuli-switchable hydrogenation/dehydrogenation catalysis for reversible hydrogen storage and delivery processes (Scheme 6f) (Semwal and Choudhury, 2016; Semwal and Choudhury, 2017). The same group has reported a series of Pd-mono and bis-imidazolylidene-NHC (Scheme 6g) and Ru-NHC-based complexes (Scheme 6h) for oxidative C-H functionalization (acetoxylation and halogenation) of arenes and oxidative scission of alkenes and alkynes to the corresponding carbonyl compounds, respectively (Desai *et al.*, 2015; Gupta *et al.*, 2016; Gupta and Choudhury, 2016).

Bera group designs and develops organometallic catalysts for the conversion of earth-abundant molecules to useful chemical feedstock. Employing ‘bifunctional’ metal-ligand cooperation strategy, new bond activation mechanism has been devised. Utilizing

water as a reagent, nitrile hydrations (Scheme 7a-b), (Daw *et al.*, 2012; Singh *et al.*, 2017) olefin oxygenation (Scheme 7c) (Ghatak *et al.*, 2015) and direct conversion of alcohol to carboxylic acid (Scheme 7d) (Sarbajna *et al.*, 2017) have been demonstrated in his laboratory. Recent research in Bera group is geared towards the development of sustainable processes and products. Alternate catalytic method is established that avoids the use of hazardous ozone generator for the selective conversion of olefins to aldehydes (Scheme 7e) (Daw *et al.*, 2014). Alcohols are catalytically dehydrogenated followed by dehydrogenative coupling on a dimetal platform (Scheme 7f) (Saha *et al.*, 2014). Employing bifunctional activation strategy, amines are doubly dehydrogenated to nitriles providing two molecules of dihydrogen under acceptorless and oxidant-free conditions (Scheme 7g) (Dutta *et al.*, 2018). This recent result raises the prospect of amines as Liquid Organic Hydrogen Carrier (LOHC). New strategies are being developed for catalyst systems that would hydrogenate the dehydrogenated product nitrile back to amine using abundant biomass. Double dehydrogenation of amine under moderate conditions with an output of two molecules of hydrogen gas and off-site refueling utilizing biomass is an important step towards the goal of establishing hydrogen economy.

Choudhury's Work

Scheme 6: Catalysts developed in Choudhury laboratory

Bera's work

Scheme 7: Bera group catalysts and their activities

Jana group at TIFR, Hyderabad demonstrated that NHC can reversibly coordinate to the *p*-block compound, diphosphene $\text{Ter}^{\text{Mes}}\text{P}=\text{P}^{\text{Ter}^{\text{Mes}}}$, at room temperature (Scheme 8) (Dhara *et al.*, 2018).

Scheme 8: Reaction of *N*-heterocyclic carbene with diphosphene

The same group further reported the *abnormal* addition of NHC^{Dip} to a conjugate acid of CAAC which leads to the adduct formation (Scheme 9)

Scheme 9: "Abnormal" addition of NHC to CAAC

(Mandal *et al.*, 2018). Mechanistic studies revealed that it proceeds through *in situ* formation of an imidazolium cation and *N*-*i*Pr substituted CAAC, followed by oxidative addition.

Concluding Remarks

It has been a challenge to capture the essence of India's organometallic chemistry and catalysis research in this concise article. The efforts have been to convey the diversity and the depth of the chemistry. Hopefully this article would allow one to assess the Indian contribution in organometallic chemistry and catalysis field. Last few years have witnessed a dramatic development in organometallic research, transitioning from classical studies on organometallic compounds to catalyst design and development for challenging molecular transformations. The infusion of young talents has made the field highly vibrant and competitive. A critical mass is reached. The future of organometallic chemistry research in India looks bright today.

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