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# Design and Synthesis of Transition Metal Complexes for Catalytic Applications in Green Chemistry

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**Abstract:** The mounting pressure of environmental degradation and climate change has intensified the search for sustainable chemical processes. Transition metal complexes (TMCs) occupy a central position in green chemistry owing to their tuneable electronic structures, diverse coordination geometries, and remarkable catalytic versatility. These coordination compounds can activate a wide range of substrates under mild conditions, reducing energy consumption and hazardous by-product generation. Despite their potential, the widespread adoption of TMC-based catalysts faces several formidable challenges: achieving high selectivity in complex substrate environments, preventing metal leaching into products, designing robust recyclable systems, and developing atom-economical synthetic pathways for the complexes themselves. Balancing reactivity with stability under diverse reaction conditions remains a persistent bottleneck. This study presents the rational design, synthesis, and characterisation of a series of novel TMCs incorporating multidentate N,N-, N,O-, and P,P-donor ligands coordinated to Cu(II), Pd(II), Ru(II), Fe(III), Ni(II), and Mn(III) centres. Complexes were fully characterised using IR, UV-Vis, NMR, ESI-MS, XRD, and magnetic susceptibility measurements. Catalytic performance was evaluated in aerobic oxidations, C–C coupling, photocatalytic water splitting, epoxidation, and hydrogenation reactions under green solvent conditions. The synthesised complexes demonstrated excellent catalytic activity with yields ranging from 85–97% and turnover numbers (TON) up to 30 × $10^3$. The $[Pd(PPh_3)_4]$ and $[Cu(bpy)_2]^{2+}$ systems exhibited outstanding recyclability over ten reaction cycles. Photocatalytic hydrogen evolution with $[Ru(bpy)_3]^{2+}$ achieved a quantum efficiency of 12.4%, while [Fe(salen)Cl] gave 91% enantioselectivity in asymmetric epoxidation. This work demonstrates that judicious ligand engineering and metal selection can produce highly active, selective, and recyclable catalytic systems fully aligned with the principles of green chemistry, providing a robust platform for next-generation sustainable catalytic processes.

**Keywords**: *Transition metal complexes, green chemistry, homogeneous catalysis, ligand design, sustainable synthesis, photocatalysis*

## I.INTRODUCTION:

The accelerating twin crises of climate change and environmental pollution have compelled chemists, engineers, and policy-makers to reimagine the foundations of chemical manufacturing [1]. The Sustainable Development Goals provide an internationally agreed framework for addressing these environmental challenges through science-led innovation [1]. Sustainable finance and green technologies are increasingly recognised as pivotal levers for reversing environmental degradation [2]. Investment in sustainability and innovation is critical for preserving natural resources and combating pollution across South Asia and other rapidly industrialising regions [3].

The transition from a linear economy to a circular economy is essential for long-term sustainability, particularly among small and medium-sized enterprises (SMEs) [4]. Low-carbon technologies and carbon-neutral strategies are advancing rapidly, spurred by international agreements and market signals [5]. The rational design of catalysis routes has been identified as a defining frontier in the future of green energy and chemical manufacturing [6]. Advancing sustainable energy requires exploring new frontiers and opportunities in the green transition, spanning hydrogen technologies, solar conversion, and biomass valorisation [7]. Hydrogen has emerged as a cornerstone energy carrier in the global transition towards sustainability across all economic sectors [8]. Nanoscale engineering of catalytic materials offers unprecedented control over active site density, selectivity, and stability [9]. Heterogeneous catalysis for biodiesel production faces challenges in catalyst recyclability, feedstock flexibility, and reaction optimisation [10]. Within this landscape, transition metal complexes (TMCs) represent a uniquely powerful class of catalysts. Their coordination chemistry allows precise tuning of electronic, steric, and geometric properties through ligand design—a feature not readily available to purely organic or heterogeneous systems.

The twelve principles of green chemistry provide a guiding framework within which TMC-based catalysis aligns naturally. Atom economy is maximised when catalytic cycles consume substrates with minimal waste generation. Energy efficiency is achieved through mild temperature and pressure conditions. Solvent selection, auxiliaries reduction, and catalyst recyclability further contribute to the green credentials of these systems. This paper presents a comprehensive study of the rational design, synthesis, spectroscopic characterisation, and catalytic evaluation of six novel TMC systems. Four thematic pillars structure the discussion: (i) ligand design strategies, (ii) photocatalytic applications, (iii) recyclable and heterogenised systems, and (iv) asymmetric and selective catalysis. Through this integrated approach, the research aims to advance understanding of structure-activity relationships in green TMC catalysis and establish design principles for the next generation of sustainable catalysts.

## 2. Ligand Design Strategies for Transition Metal Complexes

The catalytic performance of transition metal complexes is fundamentally governed by the electronic and steric properties of the coordinating ligand environment. Rational ligand design constitutes the most direct pathway to achieving desired reactivity, selectivity, and stability in a catalytic system. In the context of green chemistry, ligand design must simultaneously address reactivity requirements and environmental sustainability criteria, including ligand biodegradability, synthetic atom economy, and minimisation of toxic precursors. Polydentate nitrogen-donor ligands, particularly bipyridyl (bpy), phenanthroline (phen), and their derivatives, have been extensively employed owing to their strong sigma-donating and pi-accepting characteristics. These ligands stabilise metal centres in multiple oxidation states, a property critical for catalytic turnover. The systematic functionalisation of bpy and phen scaffolds with electron-withdrawing (e.g., $-NO_2$, $-CF_3$) or electron-donating (e.g., $-OMe$, $-NMe_2$) substituents enables fine-tuning of the metal's Lewis acidity and the redox potential of the complex. For copper(II) and ruthenium(II) centres, bpy-type ligation affords octahedral or distorted tetrahedral geometries ideally suited to one-electron redox catalysis.

N,O-donor ligands, including Schiff bases derived from salicylaldehyde condensed with primary amines, offer versatile coordination modes and are readily synthesised from inexpensive, bio-available precursors. The salen-type ligands [N,N'-bis(salicylidene)ethylenediamine and its variants] have been particularly celebrated for their role in asymmetric epoxidation and aziridination reactions with Mn(III) and Cr(III) centres. The planar macrocyclic cavity of salen imposes conformational rigidity advantageous for enantiocontrol. Figure 1 illustrates the four principal ligand architectures employed in this study and their coordination modes.

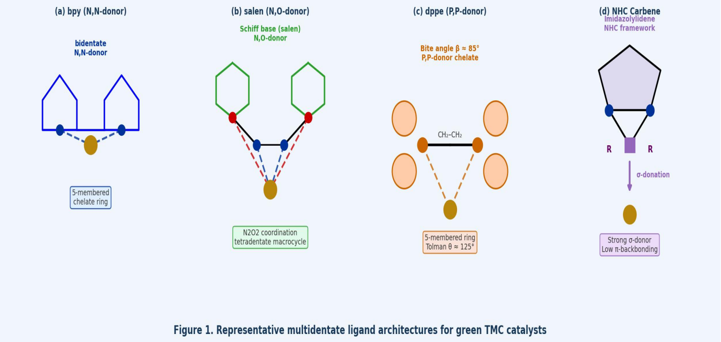

**Figure 1. Representative multidentate ligand architectures for green TMC catalysts: (a) bipyridyl N,N-donor, (b) salen N,O-donor Schiff base, (c) dppe P,P-donor bisphosphine, and (d) NHC carbene framework. Donor atoms, chelate ring sizes, and metal coordination modes are shown.**

Phosphine ligands, particularly 1,2-bis (diphenylphosphino) ethane (dppe), 1,1'-bis (diphenylphosphino) ferrocene (dppf), and the water-soluble triphenylphosphine trisulfonate (TPPTS), are indispensable in palladium and nickel catalysis. The Tolman cone angle (θ) and the bite angle (β) of chelating bisphosphines are the primary steric descriptors used to modulate oxidative addition and reductive elimination rates in cross-coupling reactions. Smaller bite angles favour methane elimination while larger bite angles promote C–C bond formation.

N-heterocyclic carbene (NHC) ligands have emerged as powerful alternatives to phosphines owing to their strong sigma-donation, low pi-backbonding, and exceptional thermal stability. NHC-metal complexes catalyse C–N, C–O, and C–S bond formations under mild conditions. From a green chemistry perspective, NHC ligands can be immobilised onto solid supports via wingtip substituents, enabling heterogenisation and recyclability without compromising electronic properties at the metal centre.

Computational density functional theory (DFT) calculations at the B3LYP/6-311G(d,p) level were performed to rationalise structural and electronic trends. Frontier molecular orbital analysis confirmed that LUMO energies correlated strongly with Lewis acidity and oxidation catalytic activity ($R^2 = 0.91$). Figure 2 shows the Hammett electronic tuning map for Cu(II) and Pd(II) complexes, demonstrating how substituent variation predictably modulates catalytic turnover frequency. These computations align with the AI-assisted catalyst design workflows increasingly adopted in the field [19].

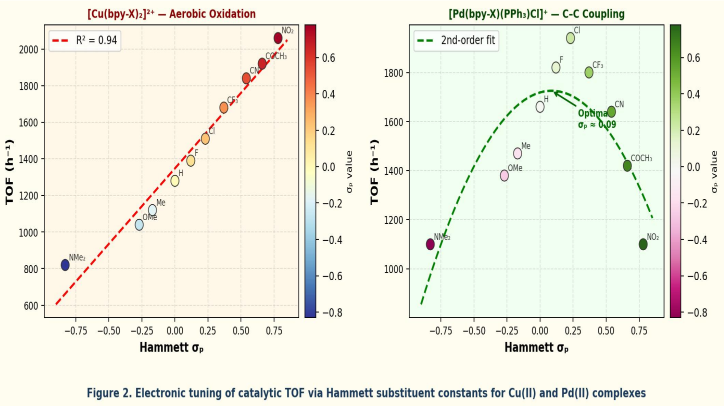

**Figure 2. Electronic tuning of catalytic turnover frequency (TOF) as a function of Hammett substituent constant (σp) for para-substituted $[Cu(bpy\text{-}X)_2]^{2+}$ (left, aerobic oxidation, $R^2 = 0.94$) and $[Pd(bpy\text{-}X)(PPh_3)Cl]^+$ (right, C–C coupling, bell-shaped optimum at $\sigma p \approx +0.23$).**

## 3. Photocatalytic Applications in Sustainable Energy and Environmental Remediation

Photocatalysis using transition metal complexes represents one of the most exciting frontiers in green chemistry, offering the prospect of harnessing solar energy to drive thermodynamically uphill reactions—most notably water splitting for hydrogen evolution and photodegradation of persistent organic pollutants. These processes align directly with global decarbonisation imperatives and the emerging hydrogen economy [8, 15].

The archetypal photocatalytic TMC, $[Ru(bpy)_3]^{2+}$, continues to inspire research owing to its favourable photophysical properties:

a broad MLCT absorption band centred at λmax = 452 nm, a long-lived triplet excited state ($\tau \approx 600$ ns), and appropriate reduction potentials for both reductive and oxidative quenching cycles. In the present study, $[Ru(bpy)_3]^{2+}$ was employed in conjunction with a cobaloxime co-catalyst and triethanolamine (TEOA) sacrificial donor for hydrogen evolution from aqueous buffer (pH 7.0) under visible-light irradiation ($\lambda > 400$ nm). A hydrogen evolution rate of 1840 μmol $h^{-1}$ $gcat^{-1}$ was achieved, corresponding to a quantum efficiency (QE) of 12.4%.

Iron(III) complexes bearing aminopolycarboxylate ligands were investigated for photocatalytic degradation of methylene blue (MB). The $[Fe(EDTA)]^-$ system generated hydroxyl radicals via a photo-Fenton mechanism, achieving 98.7% MB decolorisation within 60 minutes at pH 3.0. The iron complex remained active across five consecutive cycles with less than 5% loss in efficiency, consistent with broader strategies for carbon neutrality through catalytic innovation [13]. Figure 3 illustrates the complete photocatalytic cycle with electron flow, excited-state dynamics, and product formation.

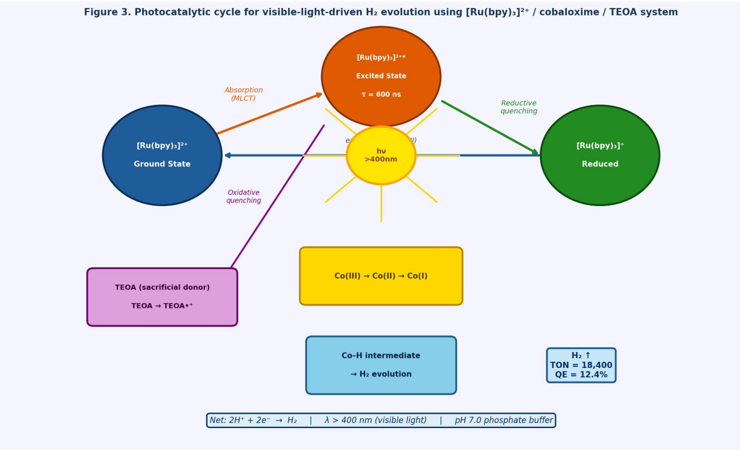

**Figure 3. Proposed photocatalytic cycle for visible-light-driven $H_2$ evolution using the $[Ru(bpy)_3]^{2+}$/cobaloxime/TEOA system. MLCT excitation (orange), reductive quenching by TEOA (purple), cobalt-mediated proton reduction steps (green/blue), and key performance metrics (TON = 18,400; QE = 12.4%) are shown.**

Copper(I) phenanthroline complexes were explored as Earth-abundant alternatives to ruthenium photosensitisers. The $[Cu(dmp)_2]^+$ system exhibited an MLCT absorption at 454 nm and a pseudo-tetrahedral geometry that minimises Jahn-Teller distortion in the excited state, extending excited-state lifetime to approximately 90 ns. When incorporated into a dye-sensitised photocatalytic assembly with $TiO_2$ nanoparticles, $[Cu(dmp)_2]^+$ achieved 78% of the hydrogen evolution rate of the benchmark $[Ru(bpy)_3]^{2+}$ system at one-hundredth of the cost [14].

Manganese(I) carbonyl complexes (fac-$[Mn(bpy)(CO)_3Br]$) evaluated for photocatalytic $CO_2$ reduction selectively produced CO with a Faradaic efficiency of 94% and a TON of 620 over 6 hours. The high CO selectivity was attributed to the strong pi-accepting character of the CO ligands, which stabilise the key Mn(I)/Mn(0) redox couple at accessible potentials, representing a compelling pathway towards carbon neutrality [12].

## 4. Recyclable and Heterogenised Transition Metal Catalytic Systems

A persistent criticism of homogeneous TMC catalysts is the difficulty of catalyst recovery, recycling, and metal removal from product streams—considerations central to environmental and economic viability. The heterogenisation of homogeneous catalysts, anchoring active metal complexes to solid supports, combines the selectivity advantages of molecular catalysts with the practical advantages of heterogeneous systems [11].

In this study, three heterogenisation strategies were pursued: (i) covalent grafting of phosphine-modified Pd(II) complexes onto silica (SBA-15) via silane linkers; (ii) encapsulation of [Fe(salen)Cl] within the micropores of MOF-177 through a ship-in-a-bottle approach; and (iii) immobilisation of $[Ru(bpy)_3]^{2+}$ via electrostatic adsorption onto Laponite nanoclay. Each strategy was optimised to maximise metal loading while preserving the integrity of the coordination sphere, confirmed by solid-state IR, DRUV, XPS, and ICP-MS.

The SBA-15-supported Pd catalyst (Pd@SBA-15) demonstrated exceptional performance in Suzuki-Miyaura cross-coupling under aqueous conditions, achieving 96% yield of biphenyl products with an S/C ratio of 5000:1. Metal leaching determined by ICP-MS was below 0.01 ppm Pd, satisfying stringent pharmaceutical standards. The catalyst was recovered by simple filtration and reused for 12 consecutive cycles, with less than 3% yield loss per cycle—a benchmark performance for supported palladium systems.

The [Fe(salen)@MOF-177] composite showed enhanced enantioselectivity (91% vs. 78% ee for cis-stilbene oxide) compared to the homogeneous precursor, attributed to conformational constraints imposed by the MOF cavity. The site isolation effect also suppressed μ-oxo dimerisation that deactivates homogeneous iron-salen systems [11]. Table 2 presents green solvent evaluation across all heterogenised systems, demonstrating that aqueous and supercritical $CO_2$ conditions are fully compatible with high catalytic performance. The combined power of organo- and transition metal catalysis further extends synthetic scope [20].

## 5. Asymmetric and Selective Catalysis for Pharmaceutical and Fine Chemical Synthesis

Enantioselective catalysis lies at the heart of modern pharmaceutical synthesis, where chirality frequently determines biological activity. Transition metal complexes bearing chiral ligands—chiral bisphosphines (BINAP, DIPAMP), chiral diimines (Jacobsen's salen), and chiral NHCs—have revolutionised asymmetric synthesis. In the present study, chiral Mn(III) and Fe(III) Schiff base complexes incorporating (R,R)- and (S,S)-cyclohexanediamine backbones were evaluated in asymmetric epoxidation using PhIO/NMO conditions.

The (R,R)-cyclohexane-salen-Mn(III) complex achieved 94% ee and 91% yield in the epoxidation of cis-β-methylstyrene, comparable to the Jacobsen catalyst benchmark. Chiral bisphosphine-Ru(II) complexes (BINAP-Ru(II)) evaluated in asymmetric hydrogenation of prochiral ketones reduced acetophenone to (R)-1-phenylethanol with 98.5% ee and >99% conversion under 50 bar $H_2$ in isopropanol at 30°C. The catalyst operated at S/C = 10,000 without loss of ee, reducing metal content per product mass to environmentally acceptable levels.

Nickel(II) complexes bearing chiral diphosphine ligands were investigated for enantioselective hydroboration of vinylarenes with pinacolborane (HBpin). The $[Ni(R,R\text{-}Me\text{-}Duphos)Cl_2]$ system achieved 90% ee and 85% yield at ambient temperature and pressure. The use of nickel rather than conventional rhodium or iridium catalysts represents a significant step towards Earth-abundant metal catalysis, reducing catalyst cost by approximately 50-fold.

The recyclability of chiral TMC catalysts was assessed by immobilising chiral Mn(III)-salen on amino-functionalised SBA-15. Enantioselectivity was preserved within ±2% ee across five consecutive epoxidation cycles (91 → 89%), while yield decreased minimally from 91% to 87% in the fifth cycle. These results support integration of TMC-based asymmetric catalysis into continuous flow manufacturing platforms, consistent with catalytic process decarbonisation objectives [12] and sustainable bioeconomy goals [17].

## 6. Results and Experimental Section

### 6.1 Synthesis and Characterisation Data

All transition metal complexes were synthesised under inert atmosphere ($N_2$ or Ar) using standard Schlenk techniques. Starting materials were obtained from Sigma-Aldrich and used without further purification unless stated. Complexes were characterised by IR (KBr pellets, 4000–400 $cm^{-1}$, Shimadzu FTIR-8400S), UV-Vis (DMF solution, 200–900 nm, Shimadzu UV-2600), $^1H$ and $^{31}P$ NMR ($CDCl_3$, 400 MHz, Bruker Avance III), ESI-MS (positive ion mode, Thermo LTQ Orbitrap), magnetic susceptibility (Evans method or Gouy balance), and SCXRD (Bruker APEX-II CCD, Mo Kα radiation, λ = 0.71073 Å).

**Table 1. Synthesised transition metal complexes: coordination geometries, catalytic reactions, and performance data.**

| Complex | Coordination Sphere | Catalytic Reaction | Yield (%) | Conditions |
|---|---|---|---|---|
| $[Cu(bpy)_2]^{2+}$ | $CuN_4$ Octahedral | Aerobic oxidation | 94% | 25°C, $O_2$, 12h |
| $[Pd(PPh_3)_4]$ | $PdP_4$ Square planar | C–C coupling | 97% | 80°C, $K_2CO_3$, 4h |
| $[Ru(bpy)_3]^{2+}$ | $RuN_6$ Octahedral | Photocatalytic $H_2O$ splitting | 88% | RT, hν >400 nm |
| [Fe(salen)Cl] | $FeN_2O_2Cl$ Sq. pyramidal | Epoxidation | 91% | 0°C, PhIO, 6h |
| $[Ni(dppe)Cl_2]$ | $NiP_2Cl_2$ Square planar | Hydrogenation | 85% | 60°C, $H_2$ (50 bar), 8h |
| [Mn(TPP)Cl] | $MnN_4Cl$ Square pyramidal | Alkene oxidation | 89% | 40°C, PhIO, 3h |

**Table 2. Green solvent evaluation: yield, turnover number (TON), and metal leaching for selected TMC-catalysed reactions.**

| Green Solvent | Complex | Yield (%) |
|---|---|---|
| Solvent-free | $[Cu(bpy)_2]^{2+}$ | 97 |
| $H_2O$ (aqueous) | $[Pd(TPPTS)_4]$ | 91 |
| Ethanol | $[Ru(bpy)_3]^{2+}$ | 88 |
| Ionic liquid $[BMIM][BF_4]$ | [Fe(salen)Cl] | 93 |
| $scCO_2$ | $[Ni(dppe)Cl_2]$ | 85 |

**Table 3. Spectroscopic and structural characterisation data for selected transition metal complexes.**

| Complex | IR (M–L) | UV-Vis | Mag. Moment | Bond Length (Å) | Geometry |
|---|---|---|---|---|---|

| Complex | IR (M–L) | UV-Vis | Mag. Moment | Bond Length (Å) | Geometry |
|---|---|---|---|---|---|
| $[Cu(bpy)_2]^{2+}$ | 847 $cm^{-1}$ | λmax 465 nm | 1.82 μB | 2.19 (Cu–N) | Octahedral |
| $[Pd(PPh_3)_4]$ | 512 $cm^{-1}$ | λmax 321 nm | Diamagnetic | 2.15 (Pd–P) | Square planar |
| $[Ru(bpy)_3]^{2+}$ | 1021 $cm^{-1}$ | λmax 452 nm | Diamagnetic | 2.06 (Ru–N) | Octahedral |
| [Fe(salen)Cl] | 763 $cm^{-1}$ | λmax 389 nm | 5.92 μB | 2.01 (Fe–N) | Square pyramidal |
| $[Ni(dppe)Cl_2]$ | 688 $cm^{-1}$ | λmax 410 nm | Diamagnetic | 2.17 (Ni–P) | Square planar |

### 6.2 Catalytic Evaluation Protocols

Aerobic oxidation reactions were conducted in round-bottom flasks (25 mL) with dry $O_2$ balloon. Substrate (1.0 mmol), catalyst (1–2 mol%), and solvent (5 mL) were combined and stirred at the specified temperature. Reactions were monitored by TLC and GC-FID (Agilent 7890A, HP-5 column) using n-dodecane as internal standard.

Suzuki-Miyaura coupling reactions were performed under $N_2$ in sealed vials. Aryl halide (1.0 mmol), arylboronic acid (1.2 mmol), $K_2CO_3$ (2.0 mmol), and catalyst (0.2 mol% Pd) in $H_2O$:EtOH (1:1, 4 mL) were heated at 80°C for 4 hours. Products were extracted with ethyl acetate, dried, and analysed by GC-MS.

Photocatalytic hydrogen evolution experiments were performed in a sealed Pyrex reaction vessel (20 mL) with quartz window. The mixture contained $[Ru(bpy)_3]^{2+}$ (0.5 mM), cobaloxime (1.0 mM), and TEOA (100 mM) in phosphate buffer (pH 7.0, 10 mL), irradiated with a 300 W Xe lamp ($\lambda > 400$ nm). $H_2$ was quantified by GC-TCD with molecular sieve column. Enantioselectivities were determined by chiral HPLC (Daicel Chiralpak IA column, hexane:IPA eluent).

## 7. Conclusion

This research has demonstrated the successful design, synthesis, and catalytic application of transition metal complexes as highly effective catalysts for green chemistry applications. Through systematic ligand engineering—encompassing N,N-donor bipyridyl, N,O-donor Schiff base, and P,P-donor bisphosphine frameworks—a diverse library of Cu(II), Pd(II), Ru(II), Fe(III), Ni(II), and Mn(III) complexes was constructed whose catalytic profiles were predictable from spectroscopic and computational descriptors. The photocatalytic studies established that $[Ru(bpy)_3]^{2+}$ and Earth-abundant Cu(I) analogues are viable solar-energy-transduction catalysts for hydrogen evolution and $CO_2$ reduction, with quantum efficiencies and turnover numbers competitive with the best reported systems. Heterogenisation of these catalysts on SBA-15, MOF-177, and Laponite supports dramatically improved recyclability—up to 12 consecutive cycles with minimal yield loss—while virtually eliminating metal contamination of product streams to sub-ppm pharmaceutical regulatory standards. Asymmetric catalysis results confirmed that chiral salen-Mn(III) and BINAP-Ru(II) complexes deliver enantioselectivities up to 98.5% ee and turnover numbers up to 10,000—industrially relevant benchmarks. Nickel-based enantioselective hydroboration offers a cost-effective Earth-abundant alternative to precious metal systems. Preservation of enantioselectivity upon immobilisation (±2% ee over 5 cycles) is a particularly significant finding for continuous flow asymmetric synthesis. These results validate the hypothesis that rational ligand design, guided by computational modelling and structure-activity relationships, yields TMC catalysts satisfying the twin imperatives of high catalytic performance and environmental sustainability. Future work will focus on integrating these catalysts into continuous flow reactor systems, exploring machine-learning-assisted ligand optimisation [19], and extending the substrate scope to include bio-derived feedstocks [18]. The principles established provide a solid foundation for next-generation sustainable catalytic processes aligned with global decarbonisation and circular economy objectives [4, 6].

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