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Research Article

Solvent-free mechanochemical synthesis and structural elucidation of novel schiff bases derived from 2-hydroxy-1-naphthaldehyde and aromatic amines

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ABSTRACT

The growing demand for environmentally benign synthetic protocols has propelled mechanochemistry to the forefront of green chemistry. This work presents a facile, solvent-free mechanochemical approach for the synthesis of 1-((4-iodophenyl)imino)methyl)naphthalien-2-ol,4-((2-hydroxynaphthalien-1-yl)methylene)amino)benzonitrile,2-((2-hydroxynaphthalen-1-yl)methylene)amino)benzonitrile,3-((2-hydroxynaphthalen-1-yl)methylene)amino)benzoic acid,4-((2-hydroxynaphthalene)amino)benzoic acid and 4-((2-hydroxynaphthalen-1-yl)methylene)amino)phenol novel Schiff bases (SB 1-6) via the condensation of 2-hydroxy-1-naphthaldehyde with selected primary aromatic amines (4-iodoaniline, 4-aminobenzonitrile, 2-aminobenzonitrile, 3-aminobenzoic acid, 4-aminobenzoic acid, and 4-aminophenol). The mechanochemical synthesis was carried out at ambient room temperature (approximately 25-30^{0}C) by simple grinding in a mortar and pestle, proceeded efficiently within 45 minutes, yielding coloured products in high yields (82.3-92.5%). The synthesized Schiff bases were characterized by (FT-IR) spectroscopy, Powder X-Ray Diffraction (PXRD), and elemental (CHN) analysis. The disappearance of primary amine and aldehyde peaks in the IR spectra, coupled with the appearance of a characteristic azomethine (C=N) stretch between 1577-1626 cm^{-1}, confirmed Schiff base formation. PXRD analysis further verified the formation of new, distinct crystalline phases, with no residual starting materials detected. This study underscores the efficacy of mechanochemistry as a rapid, high-yielding, and environmentally friendly alternative to conventional solution-based methods for Schiff base synthesis.

Keywords: 2-Hydroxy-1-naphthaldehyde, FT-IR, Green Synthesis, Mechanochemistry, PXRD, Schiff Base

Introduction

Schiff bases, characterized by the presence of an azomethine (-HC=N-) group, represent a crucial class of organic compounds first reported by Hugo Schiff in 1864 [1]. Recent reviews continue to highlight their significance as privileged scaffolds in medicinal chemistry [15, 16]. They are the condensation products of primary amines with carbonyl compounds (aldehydes or ketones) and have garnered immense interest due to their structural diversity and wide-ranging applications [17]. These compounds are extensively used as ligands in coordination chemistry [2, 18, 19], and serve as key intermediates in the synthesis of pharmaceuticals, dyes, and pigments [3, 4]. Furthermore, Schiff bases are pivotal in bioinorganic chemistry as models for developing antimicrobial, anticancer, and antioxidant agents [5, 6, 20, 21].

Conventionally, Schiff bases are synthesized in solution, often requiring prolonged heating under reflux in organic solvents. While effective, these methods suffer from drawbacks such as the use of volatile, toxic, and expensive organic solvents, which pose environmental and safety concerns [7]. The principles of green chemistry advocate for the reduction or elimination of -

hazardous substances, making the development of solvent-free synthetic methodologies a key research objective [8, 22]. Mechanochemistry, defined as the induction of chemical reactions through mechanical energy, has emerged as a powerful tool for sustainable synthesis [9, 23, 24]. This solvent-free or solvent-minimized approach offers numerous advantages, including reduced environmental impact, operational simplicity, enhanced reaction rates, and often improved yields [10, 11, 25]. Grinding reactants together in a mortar or a ball mill can efficiently facilitate reactions that traditionally require solvents [12, 26].

2-Hydroxy-1-naphthaldehyde is a highly functionalized aromatic aldehyde commonly used as a precursor for fluorescent chemosensors and coordinating ligands due to its donor oxygen and aldehyde sites [13, 27, 28]. Its condensation with various amines produces Schiff bases with significant potential in material science and medicinal chemistry.

The six aromatic amines selected for this study—4-iodoaniline, 4-aminobenzonitrile, 2-aminobenzonitrile, 3-aminobenzoic acid, 4-aminobenzoic acid, and 4-aminophenol—were chosen to provide a systematic variation in electronic and steric properties. This series includes electron-withdrawing substituents (iodo, cyano, carboxylic acid) and an electron-donating group (hydroxyl), as well as positional isomers (ortho vs. para cyano; meta vs. para carboxylic acid). This diversity allows evaluation of substituent effects on mechanochemical reaction efficiency, yield, and product crystallinity, while the functional groups (I, CN, COOH, OH) offer potential anchoring sites for metal coordination in future applications.

In this communication, we report a clean, efficient, and solvent-free mechanochemical synthesis of six novel Schiff bases derived from 2-hydroxy-1-naphthaldehyde and selected aromatic amines. The synthesized compounds were fully characterized by FT-IR, PXRD, and elemental analysis, confirming the success of this green synthetic strategy.

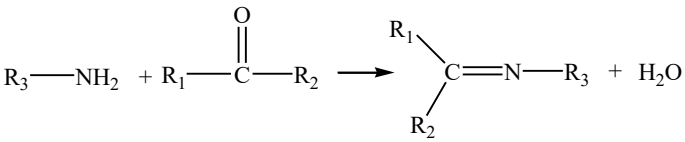
Material and Methods

2-Hydroxy-1-naphthaldehyde (Aldrich, ≥98%), 4-iodoaniline (Aldrich, ≥99%), 4-aminobenzonitrile (Aldrich, ≥98%), 2-aminobenzonitrile (Aldrich, ≥98%), 3-aminobenzoic acid (Aldrich, ≥98%), 4-aminobenzoic acid (Aldrich, ≥99%), and 4-aminophenol (Aldrich, ≥98%) were of analytical grade and used as received.

FT-IR spectra were recorded on a Perkin-Elmer FTIR Spectrum-400 spectrometer in the range of 400-4000 cm^{-1} using the ATR technique. Powder X-ray diffraction (PXRD) measurements were carried out on a PANanalytical Empyrean X'Pert Pro X-ray diffractometer with Cu Kα radiation (λ = 1.5406 Å) operating at 40 kV and 40 mA. Data were collected over a 2θ range of 5–50° with a step size of 0.026° and a scan rate of 0.067°/s. Elemental analyses (C, H, N) were determined using a Perkin-Elmer CHNS/O 2400 series II elemental analyzer. Melting points were determined using a digital melting point apparatus (MPA100).

General Mechanochemical Synthesis of Schiff Bases

The Schiff bases (SB 1 – SB 6) were synthesized using a modified mechanochemical procedure [14]. In a typical synthesis, 2-hydroxy-1-naphthaldehyde (1 mmol, 0.172 g) and the respective primary aromatic amine (1 mmol) were accurately weighed into an agate mortar. The starting materials, used as received from Aldrich, have typical particle sizes in the range of 50–200 µm as specified by the supplier. The mixture was ground manually using a pestle for 45 minutes with moderate, consistent pressure applied by the same researcher for all syntheses to maintain uniformity. The reaction progress was monitored by the formation of an intensely coloured powder. Each Schiff base was synthesized in triplicate (n = 3) to ensure reproducibility, and the average yields are reported in Table 1. The resulting solid product was collected and stored in a sample bottle for characterization. The synthetic route is illustrated in Scheme 1.



Scheme 1. Synthetic route for the mechanochemical synthesis of Schiff bases (SB 1-6).

Where R is: 4-I-C_6H_4 (SB 1), 4-CN-C_6H_4 (SB 2), 2-CN-C_6H_4 (SB 3), 3-COOH-C_6H_4 (SB 4), 4-COOH-C_6H_4 (SB 5), 4-OH-C_6H_4 (SB 6).

Results

Synthesis and Physical Properties

Table 1. Physical properties and yields of the synthesized Schiff bases

Compound	Amine Component	Colour	Molecular Formula	Melting Point (°C)	Yield (%)
SB 1	4-Iodoaniline	Yellow	$C_{17}H_{12}NOI$	180	90.4
SB 2	4-A minobenzonitrile	Yellow Orange	$C_{18}H_{12}N_2O$	197	89.6
SB 3	2-Aminobenzonitrile	Dirty Yellow	$C_{18}H_{12}N_2O$	195	82.3
SB 4	3-Aminobenzoic acid	Yellow	$C_{18}H_{12}NO_5$	190	92.5
SB 5	4-Aminobenzoic acid	Pale Brown	$C_{18}H_{13}NO_5$	198	87.5
SB 6	4-Aminophenol	Yellow Brown	$C_{17}H_{13}NO_5$	189	89.6

The six Schiff bases were successfully synthesized via a solvent-free mechanochemical grinding method. The reactions were complete within 45 minutes, yielding coloured products as detailed in Table 1 above. The high percentage yields (82.3% to 92.5%) demonstrate the efficiency of the mechanochemical approach and are consistent with recent reports on mechanochemical Schiff base synthesis, which achieved yields up to 92% [32]. The sharp melting points (180–198 °C) observed for all ligands indicate the formation of pure, crystalline compounds, comparable to previously reported 2-hydroxy-1-naphthaldehyde-derived Schiff bases [30, 31].

The lower yield observed for SB 3 (82.3%) compared to its para-substituted analogue SB 2 (89.6%) can be attributed to steric hindrance from the ortho-cyano substituent in 2-aminobenzonitrile. This observation is consistent with

systematic studies by Tigineh and Liu [29], who reported that ortho-substituted amines often exhibit reduced reactivity in mechanochemical Schiff base formation compared to their para analogues. The cyano group adjacent to the amine functionality creates steric congestion around the reaction site, hindering nucleophilic attack on the aldehyde carbonyl.

Among the other compounds, SB 4 (3-aminobenzoic acid) gave the highest yield (92.5%), suggesting that the meta-carboxylic acid group may facilitate the mechanochemical condensation through favourable electronic or solid-state packing effects

FT-IR Spectral Analysis

Table 2. Selected FT-IR data (cm^{-1}) for the synthesized Schiff bases

Compound	ν(O-H)	ν(C=N)	Other Key Bands
SB 1	3480	1603	-
SB 2	3692	1577	ν(C≡N) ~2220
SB 3	3350	1607	ν(C≡N) ~2225
SB 4	3633	1622	ν(C=O) ~1680
SB 5	3335	1626	ν(C=O) ~1675
SB 6	3361	1599	ν(O-H phenol) ~1250

FT-IR spectroscopy was used to confirm the formation of the Schiff bases. The most significant evidence was the appearance of a strong, sharp band in the range of 1577-1626 cm^{-1}, which is characteristic of the azomethine ν(C=N) stretching vibration [15]. For instance, SB 1 showed this band at 1603 cm^{-1}, while SB 6 showed it at 1599 cm^{-1} (Table 2). The absence of the characteristic ν(C=O) stretch of the aldehyde (around 1680 cm^{-1}) and the ν(N-H) stretches of the primary amine confirmed complete condensation.

The observed ν(C=N) frequencies show a clear dependence on the electronic nature of the substituents. Compounds bearing electron-withdrawing groups exhibited C=N stretches at higher wavenumbers: SB 4 (3-COOH) at 1622 cm^{-1}, SB 5 (4-COOH) at 1626 cm^{-1}. This increase can be attributed to the electron-withdrawing inductive effect (-I), which reduces electron density on the azomethine nitrogen, thereby increasing the bond order and stretching frequency [33].

In contrast, SB 6 bearing the electron-donating hydroxyl group (-OH) showed the C=N stretch at a lower frequency (1599 cm^{-1}). The +M effect of the OH group donates electron density into the aromatic ring, slightly weakening the C=N bond and lowering its stretching frequency [34].

Broad bands observed in the region 3335-3692 cm^{-1} were assigned to O-H stretching vibrations. The broadening and shift to lower wavenumbers (e.g., SB 5 at 3335 cm^{-1}, SB 6 at 3361 cm^{-1}) compared to free O-H stretches (typically ~3600 cm^{-1}) provide evidence for intramolecular hydrogen bonding between the hydroxyl proton and the azomethine nitrogen (O-H···N) [35]. This hydrogen bonding is characteristic of o-hydroxy Schiff bases and contributes to their stability.

Powder X-Ray Diffraction (PXRD) Analysis

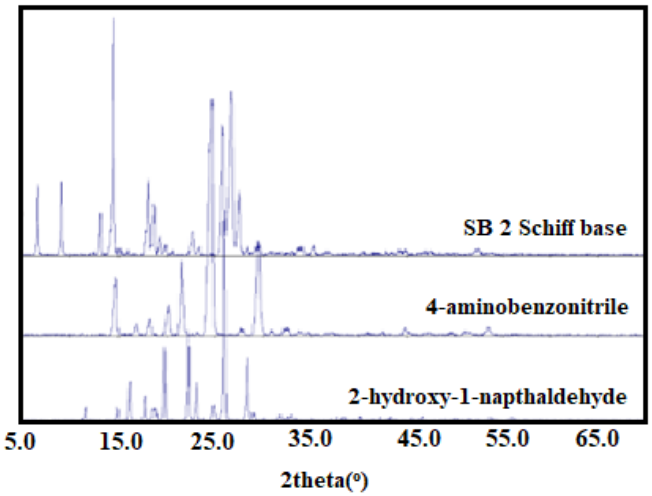


Figure 1. PXRD patterns showing the transformation from reactants to the new crystalline phase of SB 2.

The PXRD patterns of the synthesized Schiff bases were entirely different from those of the starting materials (2-hydroxy-1-naphthaldehyde and the respective amines), confirming the formation of new chemical entities. The diffractograms showed sharp and numerous reflection peaks, indicating the crystalline nature of the products. For example, SB 1 exhibited 26 reflection peaks with a maximum intensity at $2\theta = 3.42°$, while SB 2 showed 14 reflections with a maximum at $2\theta = 15.88°$. The absence of any intense peaks corresponding to the reactants in the product patterns demonstrates a quantitative conversion under mechanochemical conditions.

Elemental Analysis

Table 3. Elemental analysis results for the synthesized Schiff bases (%)

Compound	% C Found (Calc.)	% H Found (Calc.)	% N Found (Calc.)
SB 1	49.80(50.84)	2.02 (2.76)	2.95 (3.49)
SB 2	70.60 (71.88)	3.22 (3.69)	8.72 (9.31)
SB 3	70.50 (71.34)	4.24 (3.66)	8.62 (9.24)
SB 4	55.02(54.07)	3.81 (4.31)	3.59 (3.15)
SB 5	55.51(56.35)	4.84(4.02)	3.94(3.29)
SB 6	70.61(69.99)	4.24 (4.15)	4.62 (4.80)

The elemental analysis (C, H, N) results for the synthesized Schiff bases were in good agreement with the calculated values for the proposed molecular formulae (Table 3). The close correlation between the found and calculated percentages further supports the formation of the anticipated Schiff base compounds and confirms their purity.The solvent-free synthesis proceeds through several key processes [36-40]:

Mechanical energy → bond activation: Grinding generates shear and compression forces that distort crystal lattices, weakening aldehyde C=O and amine N-H bonds to facilitate nucleophilic attack [36].

Solid-state diffusion: Continuous grinding creates crystal defects and fresh surfaces, providing high-diffusivity pathways for reactant molecules to migrate and encounter each other [37].

Local temperature spike: Transient hot spots at impact points provide localized thermal energy to overcome activation barriers without bulk heating, enabling room-temperature dehydration [38].

The electron-withdrawing groups (CN, COOH) generally shift C=N stretches to higher wavenumbers, while electron-donating OH shifts to lower frequency. The ortho-CN in SB 3 introduces steric hindrance, reducing both yield and crystallinity.

Conclusions

In conclusion, we have successfully demonstrated the application of Mechanochemistry for the efficient, solvent-free synthesis of six novel Schiff bases. This green methodology offers significant advantages over conventional solution-based methods, including short reaction times (45 min), high yields (82.3-92.5%), operational simplicity, and the elimination of harmful organic solvents.

Quantitative green chemistry advantage: Compared to traditional reflux methods (typically 4-6 hours in ethanol or methanol), our mechanochemical approach achieves 5-8× faster reaction times, zero solvent consumption (saving ~20-30 mL per synthesis), and near-zero E-factor, as no waste is generated beyond the products themselves [41].

The structures of all compounds were unequivocally confirmed by FT-IR, PXRD, and elemental analysis. The successful synthesis of these ligands paves the way for their application as chelating agents in coordination chemistry—for example, forming stable complexes with transition metals such as Cu(II), Ni(II), or Co(II) through the azomethine nitrogen and phenolic oxygen donor sites [42]. These metal complexes could exhibit enhanced biological activities or catalytic properties, all within the framework of sustainable and environmentally conscious science.

Author Contributions

Conceptualization: MMH; Methodology: MMH, DAG; Investigation: MMH; Formal Analysis: MMH, DAG; Writing – Original Draft: MMH; Writing – Review & Editing: MMH, DAG; Supervision: DAG. All authors then critically reviewed the following versions. All authors have read and approved the final version of the paper and its submission and have given consent for publication.

Declaration of Generative AI and AI-Assisted Technologies in Scientific Writing

Nothing to disclose.

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Data Availability Statement

All relevant data are available within the manuscript

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Conflict of Interest

The authors declare that they have no conflicts of interest.

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