Energy-Efficient Mechanochemical Synthesis of Novel Azomethines via Sustainable Schiff Base Formation

Simnikiwe Nogqala[1], Rosalyn Klein[1], Xavier siwe-noundou[3] and Rui Krause[1]

[1]Department of Chemistry, Rhodes University, Grahamstown 6140, South Africa

[2]Pharmaceutical Sciences Department, School of Pharmacy, Sefako Makgatho Health Sciences University, Pretoria, 0204, South Africa

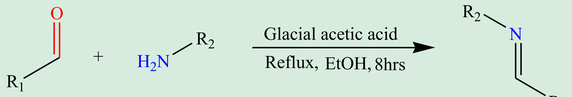
[3]Department of Chemistry, Tshwane University of Technology, Pretoria, 0001, South Africa

BACKGROUND AND MOTIVATION

Azomethines (Schiff bases) are versatile nitrogen-containing compounds with important applications in medicinal chemistry, coordination chemistry, catalysis, and materials science due to their diverse biological activities and strong metal-binding properties. Conventional synthesis typically requires organic solvents, elevated temperatures, and prolonged reaction times, generating significant waste and energy demand. Mechanochemistry has emerged as a sustainable alternative, enabling rapid, solvent free reactions through mechanical activation.

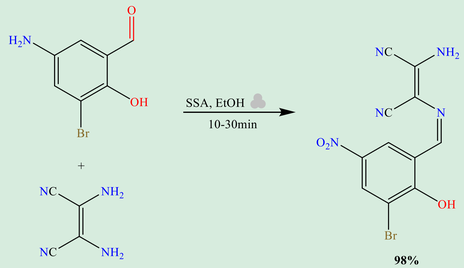
However, the integration of mechanochemistry with heterogeneous catalysis for azomethine synthesis remains underexplored. This study investigates silica sulfuric acid-catalysed mechanochemical synthesis of azomethines under planetary ball-milling conditions, providing a rapid, efficient, and environmentally benign route to Schiff-base compounds.

METHODOLOGY



Scheme 1. conventional Schiff base reaction.

This work:



Scheme 2. Mechanochemically activated Schiff base reaction using sulfuric acid immobilized silica as an additive.

Table 1. Effect of different solvents on the scope of the Schiff base reaction.

Solvent	Time (h)	Yield (%)
Methanol	9	63
THF	10	45
Ethanol	8	75

Table 2. Effect of different solvents on the scope of the Schiff base reaction.

Catalyst	Time (min)	Yield (%)
Glacial acetic acid	180	68
Silica sulphuric acid	30	83

CATALYTIC PERFORMANCE

Table 3. Effect of Catalyst Quantity on Yield

Catalyst (mg)	Time (min)[Freq.(Hz)]	Yield (%)
20	30(27)	68
40	30(27)	70
60	30(27)	75
80	30(27)	82
100	30(27)	95

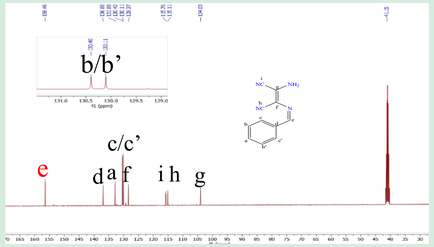


Figure 1. 13C NMR of a Schiff-base derivative

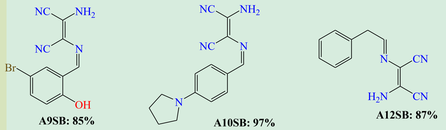


Figure 3. Schiff-base derivatives

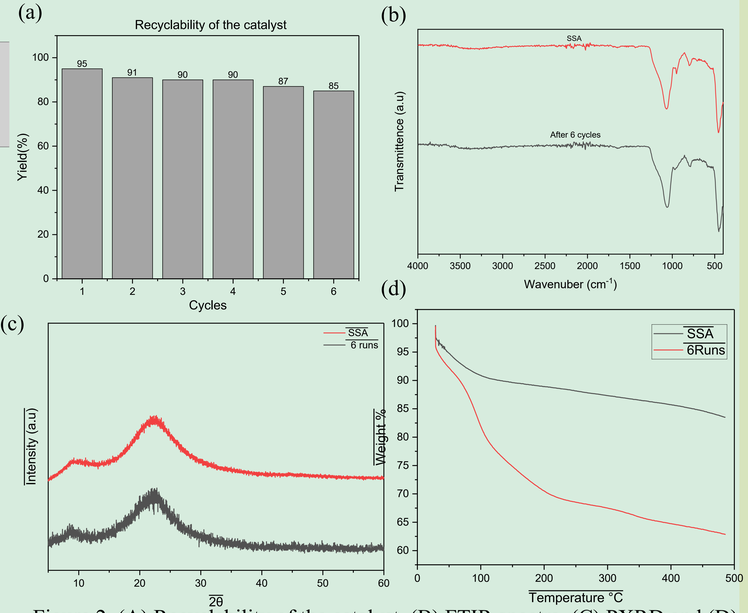
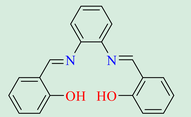
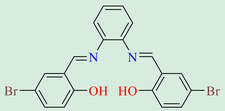


Figure 2. (A) Recyclability of the catalyst, (B) FTIR spectra, (C) PXRD and (D) TGA curves before and after 6 cycles.





ACKNOWLEDGEMENTS