

Research Article

CES as an Efficient Natural Catalyst for Synthesis of Schiff Bases under Solvent-Free Conditions: An Innovative Green Approach

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A mild and efficient method has been reported for the preparation of Schiff base ligands through the condensation reaction of various aromatic aldehydes with substituted aromatic amines in the presence of CES (calcined eggshell) as a heterogeneous catalyst under solvent-free conditions. The advantages of this ecofriendly, economic method are simplicity of the reaction procedure, moderate to good product yields, and very short reaction times.

1. Introduction

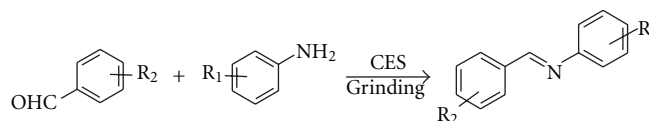
Nowadays the solvent-free approach to the synthesis of molecules becomes an attractive one since the majority of solvents are either toxic or flammable and adds considerably to the cost of overall synthesis. In many cases, the solvent-free approach improves selectivity, reduces reaction time, and simplifies separation and purification of products than the conventional methods [1–5]. The development of cleaner methods is a major challenge in green chemistry. Among the several aspects of green chemistry, the reduction or replacement of volatile organic solvents from the reaction medium is of utmost importance [6, 7]. For the increasing environmental and economical concerns in the recent years, it is now essential for chemists to search for as many environmentally benign methods as possible.

Schiff bases are an important class of ligands in coordination chemistry and find extensive application in different fields. Schiff bases derived from aromatic carbonyl compounds have been widely studied in connection with metalloprotein models and asymmetric catalysis, due to the versatility of their steric and electronic properties.

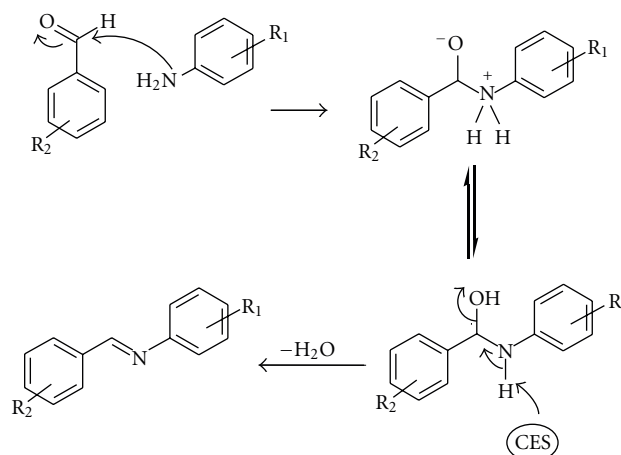
Their biologically active complexes have been often used as chelating ligands in the coordination chemistry of transition metals as radiopharmaceuticals for cancer targeting, agrochemicals, model systems for biological macromolecules, catalysts, and dioxygen carriers [8–10].

Schiff base ligands, as a variety of compounds with imine group, have gained importance because of the physiological and pharmacological activities associated with them. They constitute an interesting class of chelating agents capable of coordination with metal ions to give complexes, which serve as models for biological systems [11–13]. A lot of investigations have been made concerning the synthesis of Schiff bases [14–19], but these procedures have some limitations such as low yield, long reaction times, high environmental pollution regarding the solvent, and hardness of reaction workup. These are once again topical in connection with a diverse range of application in organic synthesis and bioorganic and medicinal chemistry. Also these complexes find many important catalytic applications, such as catalysts for epoxidation of olefins [20], alkene cyclopropanation [21], trimethylsilylcyanation of aromatic aldehydes [22], borohydride reduction of aromatic ketones [23], asymmetric oxidation of methyl phenyl sulfide [24], enantioselective epoxidation of silyl enol [25], ring-opening polymerization of lactide [26], application of ion-selective electrodes [27–31], determination of heavy metal ions in environmental samples [32], and extraction of metal ions [33].

The main component of eggshell is CaCO_3 (94–95%). During calcination, at 900°C eggshell powder decomposes due to decarboxylation and is converted into fine CES powder. About 27–28% weight loss was observed after



SCHEME 1: Synthesis of Schiff bases.



SCHEME 2: A proposed mechanism for the synthesis of a Schiff base.

calcination. Thus, CES is almost pure lime (CaO) with minor traces of magnesium oxide [34].

The first step of the mechanism during the Schiff Base formation is the nucleophilic attack of an amine on the electrophilic carbonyl carbon of aldehyde to form an imine and is a reversible step. The formation of a Schiff base from an imine largely depends on the rate of removal of water in the final step (Scheme 2). The classical synthesis of imines, originally reported by Schiff [35], involves condensation of a carbonyl compound with an amine under azeotropic distillation [36] to separate the liberated water. Subsequently, removal of water was facilitated by the use of molecular sieves [37]. Recently an *in situ* dehydration strategy has been adopted by the use of dehydrating solvents such as tetramethyl orthosilicate [38] and trimethyl orthoformate [39]. Therefore, in the present protocol, we employed CES as a dehydrating agent for the conversion of an imine into corresponding Schiff base as products.

2. Result and Discussion

The objective of present research work is to provide green methodologies for the synthesis of Schiff bases. A highly efficient and simple method has been described for the syntheses of Schiff bases with moderate to good yields.

Present syntheses comply with the principle of green chemistry. We herein report an efficient practical technique using a simple mortar and pestle method (mechanochemistry) for the synthesis of a Schiff base in presence of CES, an efficient catalyst (Scheme 1). The overall progress of the reaction was monitored by TLC and the synthesized derivatives were characterized by their IR and NMR spectra.

The reaction between a carbonyl compound and an amine leading to the formation of Schiff bases should be a

facile reaction due to the good electrophilic and nucleophilic characteristic properties of the carbonyl and amine groups, respectively.

Recently, we reported lemon juice as a natural catalyst for C–C and C=N bond formation which efficiently catalyzes the Knoevenagel and Biginelli reactions [40, 41]. In continuation with our research work using natural catalysts for organic transformations, to our satisfaction we found that the use of a stoichiometric amount of CES resulted in quantitative yield of the corresponding Schiff base at ambient temperature within 10–15 min (Table 1). However, no result was obtained when reactions were carried without employing a catalyst in all cases (except vanillin). In case of the vanillin reaction was forwarded with prolonged time with low yield. The role of CES in catalyzing the reaction was demonstrated by the lack of Schiff base formation (low yield), when the reactions were carried out in the absence of catalyst.

To establish the scope and limitations of CES as a catalyst for Schiff base formation, structurally diverse carbonyl compounds were treated with variously substituted amines such as 4-methylaniline, 4-methoxyaniline, 4-bromoaniline, 4-chloroaniline, and 4-nitroaniline under the catalytic influence of CES and the results are summarized in Table 1. Excellent results were obtained in most cases.

The treatment of vanillin with 4-methylaniline and 4-methoxyaniline afforded the corresponding Schiff bases in greater than 94% yields in 10–15 min (Table 1, entries 5 and 10), while the yield was reduced when it was treated with 4-bromoaniline and 4-chloroaniline (Table 1, entries 15 and 20) and a very poor (40%) yield was obtained when vanillin was treated with 4-nitroaniline (Table 1, entry 25). The quantitative yield was obtained during the reaction of

4-methylaniline and 4-methoxyaniline with 4-nitrobenzaldehyde in 10 min (Table 1, entries 3 and 8) in the presence of the catalyst.

It was also observed that a low yield (59%) was resulted for the reaction of 4-methoxybenzaldehyde with 4-nitroaniline (Table 1, entry 22), as in the case of 4-methoxybenzaldehyde, the electrophilicity of the carbonyl carbon reduces due to the presence of the electron-donating substituent group through resonance while the strong electron-withdrawing substituent group of 4-nitroaniline decreases its nucleophilicity.

While again, the less nucleophilic amines such as 4-bromoaniline and 4-chloroaniline were treated with less electrophilic aldehydes such as 4-methoxybenzaldehyde (Table 1, entries 12 and 17) and 4-N,N-dimethylbenzaldehyde (Table 1, entries 14 and 19), moderate results were obtained in all cases. It was also observed that if one of the reactants is deactivated by its substituent, a comparatively more reaction time is required to complete the reaction checked by TLC.

The comparison of the results obtained by our method with some of the reported catalysts for synthesis of Schiff bases (Table 2) shows the efficiency of this method due to good yield, shorter reaction time, and application of an inexpensive catalyst prepared from a readily available natural material.

3. Experimental Section

3.1. General. All the chemicals were of commercial reagent grade. The aromatic aldehydes and amines were purified by standard procedures. Melting points were measured on open capillary method and on DBK-programmable melting point apparatus. IR spectra were recorded in KBr pellets on the Bruker ALPHA FT-IR spectrometer. $^1\text{H-NMR}$ spectra were measured with Avance-300 F-NMR instrument using CDCl_3 as a solvent and TMS as an internal standard. The purity determination of the substrates and reaction monitoring were accomplished by TLC using Merck silica gel 60 F₂₅₄ plates.

3.2. Preparation of Catalyst CES. Chicken eggshells were collected in bulk and cleaned thoroughly with distilled water. The broken shells are then boiled in water to remove any adhesion. After cleaning, eggshells are dried in oven at 80°C and crushed into fine powder using mortar and pestle. This raw eggshell powder was then calcinated at a heating rate of 2°C/min in Muffle furnace to 900°C and this temperature was maintained for 3 hrs. The thermal treatment had two parts: in the first 30 minutes, most of the organic materials were burnt out, whereas in the second part the eggshell get transformed into white soft powder which was denominated as CES.

3.3. Synthesis of 4-Methoxy-N-aniline[(4-hydroxyphenyl)methylidene]6. The synthesis of 6 is described as a representative example. The reaction mixture of 4-methoxyaniline (0.123 g, 1 mmol), 4-hydroxybenzaldehyde (0.122 g, 1 mmol), and 25 mg calcined eggshells (CES) was ground

TABLE 1: Physical characterization of Schiff bases.

Entry	Substrates		Yield%	m.p. °C Found (reported)
	R ₂ (aldehyde)	R ₁ (amine)		
1	4-OH	4-CH ₃	89	212–214
2	4-OCH ₃	4-CH ₃	90	93–95
3	4-NO ₂	4-CH ₃	98	121–125 (128–130) [42]
4	4-N(CH ₃) ₂	4-CH ₃	90	90–94
5	2-OH,3-OCH ₃	4-CH ₃	94	210–212
6	4-OH	4-OCH ₃	92	215–220
7	4-OCH ₃	4-OCH ₃	88	142–145
8	4-NO ₂	4-OCH ₃	98	68–72
9	4-N(CH ₃) ₂	4-OCH ₃	89	132–135
10	2-OH,3-OCH ₃	4-OCH ₃	96	110–112
11	4-OH	4-Br	81	188–189
12	4-OCH ₃	4-Br	80	116–118
13	4-NO ₂	4-Br	91	172–175 (176–178) [42]
14	4-N(CH ₃) ₂	4-Br	86	189–192
15	2-OH,3-OCH ₃	4-Br	86	118–119
16	4-OH	4-Cl	83	230–232
17	4-OCH ₃	4-Cl	80	90–94 (92) [42]
18	4-NO ₂	4-Cl	86	110–117
19	4-N(CH ₃) ₂	4-Cl	81	178–182
20	2-OH,3-OCH ₃	4-Cl	82	108–110
21	4-OH	4-NO ₂	65	160–162
22	4-OCH ₃	4-NO ₂	59	69–71
23	4-NO ₂	4-NO ₂	81	112–116
24	4-N(CH ₃) ₂	4-NO ₂	84	178–182
25	2-OH,3-OCH ₃	4-NO ₂	40	146–149

thoroughly using mortar and pestle. During grinding, the powdered reaction mixture was transformed into a semisolid paste after 2–3 min. The grinding was continued for 10–15 min (monitored by TLC). The reaction mixture was placed at room temperature for appropriate time till moisture was removed and get converted into dry powder. Further the resulting dry powdered yield was transferred into 50 mL water, and neutralized with dilute HCl. The product was separated by filtration, washed thoroughly with water and dried. The crude product was then purified by crystallization from ethanol and the pure Schiff base 6 was obtained.

Yield 92%; mp 215–220°C; IR (KBr): 3315 (OH), 1632 (C=N) cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3): 3.7 (s, 3H, $-\text{OCH}_3$), 7.0 (m, 2H, Ar-H), 7.2 (m, 4H, Ar-H), 7.4 (m, 2H, Ar-H), 8.4 (s, 1H, =CH), 12.9 (s, 1H, $-\text{OH}$).

The experimental procedure was followed for appropriate time to synthesize all the products listed in Table 1.

4. Conclusion

Herein, we are reporting a new ecofriendly grinding method for the synthesis of Schiff bases catalyzed by CES with a

TABLE 2: Comparison of CES with different reported methods used for synthesis of Schiff bases.

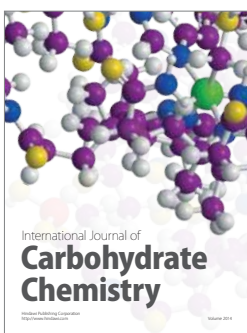
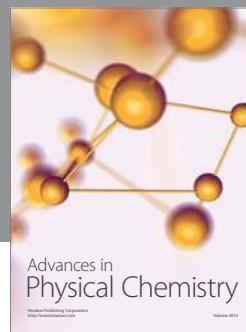
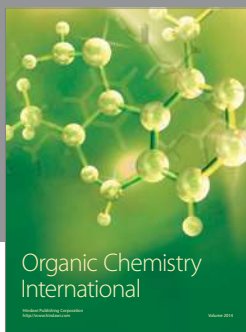
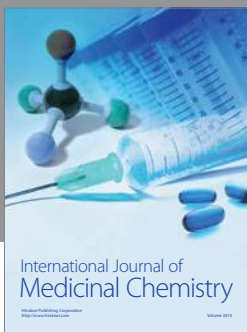
Serial no.	Catalyst	Solvent	R (R-NH ₂)	R' (R'-CHO)	Reaction condition	Time	Yield%
1	Montmorillonite [42]	Chloroform	H	H	RT grinding	10 min	95
2	CeCl ₃ ·7H ₂ O [43]	Ethanol	H	2-OH	Reflux	2 hrs	68
3	P ₂ O ₅ /Al ₂ O ₃ [44]	—	H	H	RT stirring	20 min	80
4	Mg(ClO ₄) ₂ [45]	DCE	4-NO ₂	4-OMe	RT	8 hrs	95
5	Acid catalysis [46]	Trimethyl orthoformate	—	—	Stirring	8 hrs	—
6	Alumina [47]	—	H	H	Stirring 20°C	2 hr	99
7	Acetic acid [48]	—	H	—	Grinding	—	89
8	Conc. H ₂ SO ₄ [49]	Ethanol	—	—	Reflux	1 h.	70
9	Montmorillonite K-10 clay [50]	—	H	H	MW	3 min	98
10	CES	Solvent-free	4-OCH ₃	4-NO ₂	RT grinding	10–15 min	98

moderate to good yield. The products can be purified by recrystallization using appropriate solvents. Compared with traditional methods, this solvent-free approach is more convenient, cleaner, and safe, does not employ any toxic materials, and involves mild reaction conditions and simple workup which results in maximum efficiency.

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