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Polyethene Glycol (PEG-400): An Efficient and Eco-friendly Catalyst For The Preparation of N-benzylideneaniline by Schiff base Reaction

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ABSTRACT

Schiff base compounds are gaining importance daily in this scenario due to their various biological and catalytic activities. In this study, an efficient and eco-friendly synthesis of N-benzylideneaniline was carried out in polyethene glycol (PEG-400) as a greener medium at room temperature. PEG400 was inexpensive and non-toxic, providing a high yield and efficient medium for the synthesis of Schiff bases with excellent outcomes. Furthermore, FT-IR spectroscopy is used to characterize the newly created Schiff base. The recyclability of the catalyst was also studied up to six cycles.

Keywords: Schiff base, Polyethene glycol (PEG400), Benzylideneaniline, Recyclable catalyst.

INTRODUCTION

The study of Schiff base metal complexes has become more critical in chemistry. Schiff bases are the compounds containing the azomethine group (-HC=N-). Hugo Schiff initially described them in 1864 as the condensed products of aldehydes or ketones[1]. Schiff bases have a wide range of applications in biology, "Many of these include ant-tubercular, antibacterial, antifungal, antitumor, diuretic insecticidal, herbicidal, anthelmintic anti-HIV, anticonvulsant, antihypertensive, and antiparasitic"[2,3]. Schiff's base derivatives have been studied extensively and used in various applications for over a century, including magneto chemistry, nonlinear optics[4], photophysical research[5], catalysis, materials chemistry, chemical analysis, and oxygen absorption and transport[6]. A wide variety of Schiff base Zn(II) complexes have been shown to be useful in the synthesis of electroluminescent materials. "Oxidative cyclohexane to cyclohexanol reactions are catalyzed by the use of Ru(III), Fe(III), and Co(II) complexes of Schiff bases produced from hydroxybenzaldehyde[7]. A catalytic precursor for olefin oligomerization may be generated from cyclohexanone with H_2O_2 and Ni(II) complexes with bivalent (N-N) ligands"[8]. The dye and polymer industries are said to benefit from Schiff bases. It has been claimed that chromium and cobalt complexes of Schiff base may colour leather, food packaging and wool. Its nickel complex (Ni(II)-2L) was also synthesized and studied for its properties. "In the case of 5-diethylamino-2, the two most common phenolic compounds are 5-diethylamine-2 and 5-diethylamino-2-(dimethylamino)phenylamine-2. Along with their metal(II) complexes [Cu(L1)2] (1), (2), (3), and (4) functioning as anticancer agents were synthesized as novel Schiff base ligands"[9]. Co/Ni/Cu/Zn(II) Synthetic Schiff base complexes formed from furfural-MAP and 6-methyl-2-aminopyridine were produced[10]. However, it was observed that longer reaction times, high temperatures, low yields, and the use of carcinogenic media are some of the drawbacks of the reported methods. Therefore, some efforts are made by chemists to modify the synthesis of Schiff bases by employing an efficient synthetic route using green tools.

In the last decade, solvents have increased significantly. To synthesize compounds in chemical processes, selecting an appropriate solvent and catalyst is essential. During the past few years, considerable attention has been received to various organic syntheses towards polyethene glycol (PEG) because it is non-toxic, cost-effective, readily available, nonflammable, nonvolatile and has high stability and has a safe character[11]. Moreover, PEG remains neutral in the system, due to which various functional groups remain undisturbed that is either acidic or basic. For these reasons, PEG is considered environmentally susceptible. Thus, has for different chemical transformations, PEG-400 emerged as an efficient catalyst. Furthermore, PEG-400 enhanced the reaction rate by forming strong hydrogen bonds with the substrate. As a result, the PEG-400 is widely used in many organic reactions to convert oxiranes to thiranes, asymmetric aldol condensation reactions, cross-coupling reactions, and reaction media for mono-bromination aromatics using NSB, synthesis of derivatives of triazole and pyrazole[12]. We have developed an efficient and environmentally benign synthesis of N-benzylideanilines using PEG400 as a catalyst, considering PEG's synthetic utility[13].

EXPERIMENTAL
MATERIALS AND METHODS

Materials

No purification was performed on any chemical reagents. Benzaldehyde, aniline and its derivatives, hexane, diethyl ether, toluene, DCM, ethyl acetate, magnetic stirrer, PEG-400 etc. For characterization of compounds, TLC, Bruker FT-IR Spectrometer, and Bruker advance neo 500MHz NMR Spectrometer are used.

General Procedure
Sample Preparation
Preparation of Schiff base and their derivatives

Initially, benzaldehyde (1.0 mmol) is added to PEG-400 (10 µL), and then aniline (1.0 mmol) is added to the reaction. The reaction mixture was agitated for 45 min at room temperature using a magnetic stirrer. After that, the product is monitored by TLC. The desired outcome is crystallized by hexane and ethyl acetate into ice. The product's solid form, i.e., Schiff base (N-benzylideaniline), is filtered and stored for characterization. The desired product was isolated in 98% yield. Similarly, following this method, we have done reactions between derivatives of aniline and Benzaldehyde.

Detection Method
FT-IR of PEG-400 and *N*-benzylideneaniline

The FT-IR spectra of fresh PEG-400 and recoverable PEG-400 were compared. Both show almost the same absorption peaks. When it comes to bending and stretching, polyethene glycol has you covered. In the 3446 cm^{-1} area, the OH stretch is turning[14-16]. It was a bending vibration of CH_2, causing absorption of around 1483 cm^{-1}. The C-O stretching vibration reveals a significant activity band at 1348 cm^{-1} to 1247 cm^{-1}. C-C stretching leads to a sharp bands of 975 cm^{-1} and 868 cm^{-1} [18]. The C=N extension of N-benzylideneaniline appears at 1624 cm^{-1}, and the C=C stretching band seems at 1589 cm^{-1} (Figure 2).

^{1}H NMR of N-Benzylideneaniline

Yellowish Solid, m.p. 51°C, yield: 98%, ^{1}H NMR (δH, ppm): 8.28 (1H), 7.76 (2H), 7.75-7.29 (7H) and 7.10-7.07 (1H).

^{13}C NMR (δ_C, ppm): 160.5, 152.2, 136.3, 131.5, 129.4, 126.1, 121.00, 118.6, 115.2

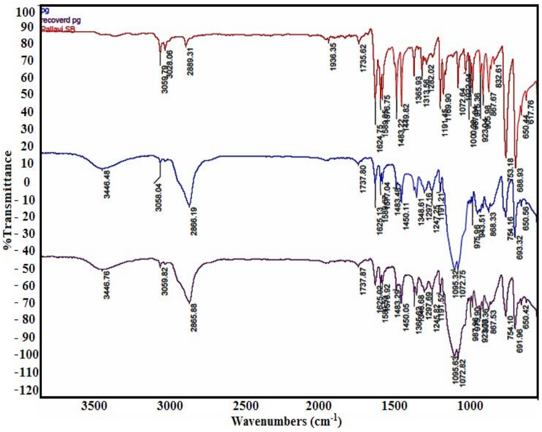


Fig. 1. FTIR Spectrum of PEG-400 and N-Benzylideneaniline

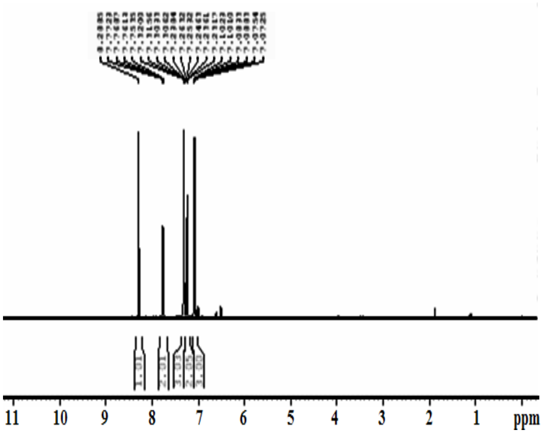


Fig. 2. ¹H NMR spectrum of Schiff's base

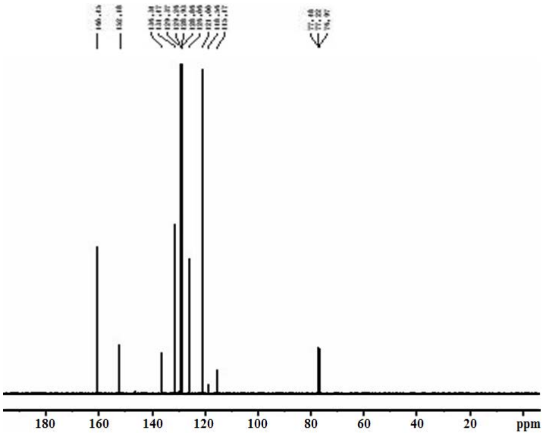
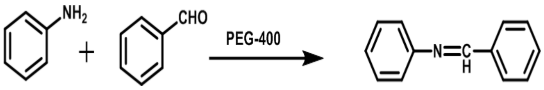


Fig. 3. ¹³C NMR spectrum of Schiff's base

RESULTS AND DISCUSSION

In our model reaction, N-benzylideaniline is synthesized using benzaldehyde and aniline (Scheme 1). Such a reaction is catalyzed by polyethene glycol (10 µL), providing a 98% yield in less than 1 h of the relative compound under neat conditions (Table 2, Entry 1). The reaction of benzaldehyde with aniline was examined in different solvents such as hexane, diethyl ether, toluene, DCM, and PEG (400)[19]. The comparisons and results are shown in Table 1. The examination shows that in solvents like hexane, diethyl ether, toluene and DCM, the product yield varies from 65%, 60%, 70%, and 68%, respectively (Table 1). Among the tested solvents, it is concluded that the reaction in PEG-400 is more facile and proceeds to give a good yield (98%) at room temperature in neat conditions[20].



Scheme 1. Schiff's base reaction using benzaldehyde and aniline

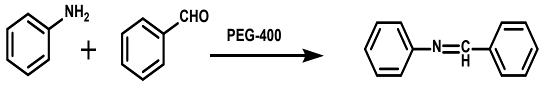


Table 1: Optimization for the synthesis of N-benzylideneaniline

Entry	Catalyst	Solvents	Reaction time (minutes)	Yield(%)
1	30 µL	Hexane	30	65
2	30 µL	Diethyl ether	30	60
3	30 µL	Toluene	30	70
4	30 µL	DCM	30	68
5	30 µL	Neat	30	85
6	20 µL	Neat	35	90
7	10 µL	Neat	40	98
8	Without Catalyst	Neat	60	35

To determine the full extent of the shift, the reaction of various derivatives of benzaldehyde with multiple products of aniline is carried out with optimized reaction conditions (Table 2). The effect of electron-withdrawing and electron-releasing substituents is also observed in the synthesis of N-benzylideaniline[21]. Aniline gives a higher yield of the relative compound with electron releasing derivatives like 4-Me, 4-OMe, and 4-OH with 95%, 80%, and 72% (Table 2, entries 2,3 and 4) yields of the relative compound, respectively. Simultaneously, electron-withdrawing groups like 4-NO_2 and 4-Cl give 85% and 90% (Table 1, entries 5 and 6) of the comparative compound[22].

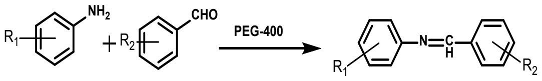


Table 2: Preparation of N-benzylideneaniline and their derivatives by Schiff base reaction[23] with PEG-400

Entry	R_1	R_2	Yield(%)
1	H	H	98
2	H	4-OH	72
3	H	4-CH_3	95
4	H	4-OCH_3	80
5	H	4-NO_2	85
6	H	4-Cl	90
7	4-OH	H	85
8	4-OH	4-OH	75
9	4-OH	4-CH_3	95
10	4-OH	4-OCH_3	75
11	4-OH	4-NO_2	90
12	4-OH	4-Cl	70
13	4-Br	H	98
14	4-Br	4-OH	80
15	4-Br	4-CH_3	85
16	4-Br	4-OCH_3	65
17	4-Br	4-NO_2	78
18	4-Br	4-Cl	70
19	4-NO_2	H	85
20	4-NO_2	4-OH	60
21	4-NO_2	4-CH_3	80
22	4-NO_2	4-OCH_3	90
23	4-NO_2	4-NO_2	60
24	4-NO_2	4-Cl	95

Recyclability test

The PEG-400 in the reaction mixture was recovered[24] and reused with no loss of activity. The recyclability of the PEG-400 was seven times greater than previously thought (Figure 4).

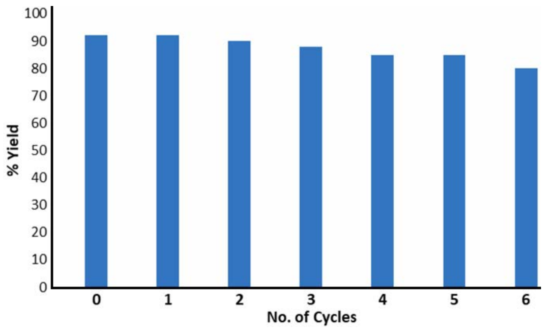


Fig. 4. Recyclability of catalyst

CONCLUSION

Schiff base is used as an active pharmaceutical agent[25]. A Schiff base reaction was synthesized using benzaldehyde and aniline with an excellent yield (98%). PEG-400 catalyzed the response. The catalyst can be recyclable. The derivatives of the Schiff base reaction were also synthesized. The desired product is confirmed using FT-IR and NMR spectroscopy.

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Conflicts of Interest

The authors declare no conflicts of interest.

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