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Synthesis of amino alcohol Schiff bases under microwave irradiation without solvent

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ABSTRACT

Chiral amino alcohol Schiff bases are important ligands widely used in asymmetric catalysis. A series of Schiff bases were synthesized under microwave without solvent. Excellent yield within short reaction time is obtained along with other advantages like mild reaction condition, eco-friendly and working-up easily. The effects of reaction time and microwave power variation on yield are observed.

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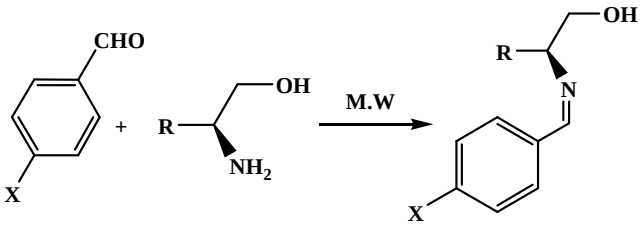
KEYWORDS

Schiff bases;
Microwave;
Solvent free;
Amino alcohol.

INTRODUCTION

Schiff bases, widely used in organic synthesis chemistry, had been known to chemists for a long time. The synthesis and derivatization of amino alcohol Schiff bases are of both pharmaceutical and chemical interest. In recent years, chiral Schiff bases have been extensively studied. Schiff bases are important intermediates for the synthesis of various bioactive products[1], and they are also known to have biological activities such as antimicrobial[2], antifungal[3], antitumor[4] and herbicidal[5] activity. On the other hand, Symmetric and asymmetric transition metal complexes of Schiff bases have been developed and used as ligands/catalysts in many reactions such as epoxidation[6], asymmetric synthesis[7], asymmetric sulfoxidation[8], asymmetric silylcyanation[9] and many other applications[10-12].

Microwave had been widely used in organic synthesis[13-15]. It has been shown to lead to dramatically reduce reaction times, increased product yields and enhanced product purities by reducing unwanted side reactions compared to the conventional method of heating[16-18].



R= CH_2Ph: 1; R= Ph: 2; X= a, H; b, OMe; c, Cl; d, Br; e, NMe_2

Scheme 1 : The synthesis of Schiff bases

EXPERIMENTAL

All reactions were performed in a commercial domestic microwave oven (Galanz P70D20P-TD). TLC was used to monitor the reaction process. TLC was GF254 thin layer chromatography with petroleum ether/ ethyl acetate (6/1) used as eluent. Melting points were determined on a microscopy apparatus and uncorrected. IR spectra were recorded on a FTIR-8400S

spectrometer (KBr). The products were also characterized by comparison of their Melting points with the literature values.

General procedure for the preparation of Schiff base (compound 1a, TABLE 1)

A mixture of L-Phenylalaninol (1.51g, 10mmol), benzaldehyde (1.02g, 10mmol) was taken and triturated in a mortar pestle. Then above mixture was transferred to a 25mL conical flask which was then kept in microwave for synthesis. Microwave was run at 119-462 W for different time. The progress of the reaction was monitored by TLC. Upon completion, the product was cooled and recrystalized with n-hexane and ethanol. White solid, m.p. 78-80°C, IR(KBr) νcm^{-1}: 3221(O-H), 2917(-CH_2-), 2816(-CH_2-), 1642(C=N), 1578(C=C), 1495(C=C), 1052(C-O). Power, time in microwave assisted synthesis and % yield are given in Figure 1.

Some other results were shown as follows:

Compound **1b**: white solid, m.p. 80-81.5°C, IR(KBr) νcm^{-1}:3213(O-H), 2920(-CH_2-), 2857(-CH_2-), 1644(C=N), 1606(C=C), 1496(C=C), 1253(=C-O-C), 1048(C-O).

Compound **1c**: white solid, m.p. 74-75°C, IR(KBr) νcm^{-1}: 3213(O-H), 2917(-CH_2-), 2854(-CH_2-), 1641(C=N), 1594(C=C), 1491(C=C), 1049(C-O), 748(-Cl).

Compound **1d**: white solid, m.p. 64-66°C, IR(KBr) νcm^{-1}: 3566(O-H), 2923(-CH_2-), 2874(-CH_2-), 1646(C=N), 1585(C=C), 1488(C=C), 1068(C-O), 698(-Br).

Compound **1e**: white solid, m.p. 120.5-121.5°C, IR(KBr) νcm^{-1}: 3323(O-H), 2921(-CH_2-), 2858(-CH_2-), 1644(C=N), 1605(C=C), 1495(C=C), 1420(-NMe_2), 1058(C-O).

Compound **2a**: white solid, m.p. 69-70°C, IR(KBr) νcm^{-1}: 3253(O-H), 3027(C-H), 2943(-CH_2-), 2855(-CH_2-), 1635(C=N), 1600(C=C), 1507(C=C), 1045(C-O).

Compound **2b**: white solid, m.p. 88-90°C, IR(KBr) νcm^{-1}: 3200(O-H), 3020(C-H), 2937(-CH_2-), 2868(-CH_2-), 1638(C=N), 1605(C=C), 1506(C=C), 1263(=C-O-C), 1070(C-O).

Compound **2c**: white solid, m.p. 61-63°C, IR(KBr) νcm^{-1}: 3254(O-H), 3031(C-H), 2956(-CH_2-), 2880(-CH_2-), 1645(C=N), 1596(C=C), 1505(C=C), 1085(C-O), 761(-Cl).

Compound **2d**: white solid, m.p. 71-72°C, IR(KBr) νcm^{-1}: 3244(O-H), 3030(C-H), 2929(-CH_2-), 2847(-CH_2-), 1646(C=N), 1587(C=C), 1488(C=C), 1064(C-O), 533(-Br).

Compound **2e**: white solid, m.p. 65-67°C, IR(KBr) νcm^{-1}: 3144(O-H), 3027(C-H), 2849(-CH_2-), 1657(C=N), 1606(C=C), 1505(C=C), 1418(-NMe_2), 1061(C-O).

RESULT AND DISCUSSION

In conventional method, Schiff bases were synthesized by condensation of amino alcohol with different substituted benzaldehydes (1:1 mole ratio) in methanol at 65°C. On the other hand, in microwave irradiation method, reactant mixtures were subjected to microwave power. As shown in TABLE 1, all condensation of amino alcohols with substituted benzaldehydes, not only be the electron-donating groups such as –$N(CH_3)_2$ and -OCH_3, but also the electron-withdrawing groups such as -Cl, -Br, under microwave irradiation at the absence of solvents have excellent yields.

As shown in Figure 1, with increase in power supply from 119W to 462W, %yield was increased at a faster rate between 119W-280W and the rate became slower above 280W. The most suitable condition was 2 minutes under the power of 280 watt. The causation may be that with the increment of time and the higher power,

TABLE 1 : Condensation of amino alcohols with substituted benzaldehydes

Compound	Time /min	Yield/%		m.p./℃	
		M.W	General	Find	Report[19,20]
1a	2	98	91	81-82	78-80
1b	2	98	66	80-81.5	78-79
1c	2	95	89	74-75	73.5-74.5
1d	2	97	78	64-66	65-67
1e	2	98	77	120.5-121.5	119.5-120.5
2a	2	98	83	69-70	70-71!
2b	2	96	92	88-90	88.5-89.5
2c	2	95	70	61-63	60-63
2d	2	93	63	71-72	71-73
2e	2	96	80	65-67	65-65.5

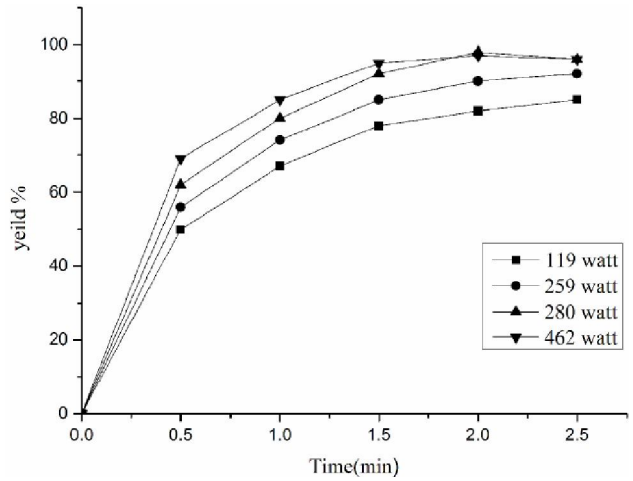


Figure 1 : %Yield vs. time at different power levels for compound 1a

the temperature is too high to make the reaction effectively carry through; On the other hand, if the power is too low or the reaction time is too short, the reaction is unable to progress completely, so the yield is too low.

CONCLUSION

In summary, compared with the conventional method, the microwave promoted condensation of amino alcohols with substituted benzaldehydes with excellent yields in very short reaction time. The solvent-free condition was eco-friendly and working-up easily.

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