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An Overview: Using Different Approaches to Synthesis New Schiff Bases Materials

Anahed A. Yaseen [1], Emaad T. B. Al-Tikrity [1], Mohammed H. Al-Mashhadani [2], Nadia Salih [3], Emad Yousif [2]

[1] Department of Chemistry, College of Science, Tikrit University, Tikrit, Iraq
[2] Department of Chemistry, College of Science, Al-Nahrain University, P. O. Box: 64021, Baghdad, Iraq
[3] Department of Chemical Sciences, Faculty of Science and Technology, Universiti Kebangsaan Malaysia, 43600 UKM Bangi, Selangor, Malaysia;

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A B S T R A C T

Schiff base compounds are called relative to the scientist who first prepared them (Hugo Schiff). They are synthesized by the condensation reaction of the carbonyl group –C=O– of the aldehyde or ketone compound with a primary amine. This leads to the formation of azomethine or imine group –C=N– plus water molecule. Schiff base molecules have gained special importance due to their biological activity, such as anti-inflammatory and antibiotic. Schiff base compounds are also utilized in the industry as corrosion inhibitors, dyes, and photo-stabilizers of plastic polymers. This short review includes highlighting of recent approaches of synthesis novel Schiff base molecules. It also discusses the mechanism of the reaction and why it is a reversible condensation reaction.

1. INTRODUCTION

Schiff bases are named relative to scientist Hugo Schiff who first synthesized it. Schiff base functional group is formed by the condensation reaction of the carbonyl group with primary amine resulting double bond between carbon and nitrogen atoms(–C=N–). Schiff base group is also known as an azomethine functional group or an imine functional group [1-3]. Compounds holding Schiff base groups are considered one of the most important organic compounds because of their wide range of applications in medications, industry, analytical chemistry, and biology [4-7]. In pharmacy and medicine fields, imine groups have gained significant attention due to their biological reactivity, such as analgesic, anti-inflammatory [8], antimicrobial [9-10], anticancer, anthelmintic, anticonvulsant, antioxidant, etc. [11-16]. The formation of hydrogen bonds between the nitrogen atom of imine functional group and the active cell center enhances the biological activity of Schiff base molecules [17-18].

Schiff base compounds are also utilized as photo-stabilizers of plastic polymers [3], catalysts of organic reactions, and inhibitors of corrosion [19].

*Corresponding author at Department of Chemistry, College of Science, Al-Nahrain University, Baghdad, Iraq;
E-mail address: emad_yousif@hotmail.com

2. Synthesis of Schiff base

The classical method for synthesis of imines is by mixing equimolar quantities of aldehyde or ketone with the primary amines, as shown in Figure 1 [20]. The reaction of imine formation is reversible, and it produces a water molecule. The reaction of aromatic ketones with primary amine is required harsh conditions such as catalyst, the longer time, and greater temperature compared with an aliphatic aldehyde, which happens spontaneously in the almost reactions. Several catalysts have been utilized to perform this reaction, such as $POCl_3$, BF_3, and acids [21].

The general reaction scheme for the synthesis of Schiff base is as follows:

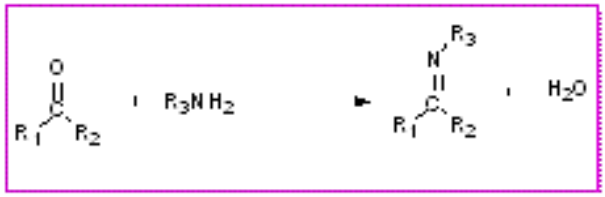

Fig 1. Schiff base (imine).

3. Novel methods to synthesize Schiff base

Interestingly imine compounds have a different type of application in both medicine and industry. Hence chemists have spent a lot of time exhibiting excellent methods of synthesizing novel materials containing imine group.

4. Utilizing glacial acetic acid and ethanol as a catalyst

In one research, some novel sulfonamides were used to synthesize Schiff bases and demonstrated several biological activities. Many Schiff bases of sulphonamides were synthesized *via* reacting an aromatic aldehyde with 4-amino benzene sulfonamides at 60 ºC in the existence of glacial acetic acid as a catalyst, as shown in Figure 2. The majority of compounds synthesized showed good antimicrobial and antifungal activity against selected strains of bacteria and fungi, namely *B. subtilis, S. aureus, E. coli*, *S. typhi, C. albicans, and A. niger* at higher concentration (200µg/ml)[22].

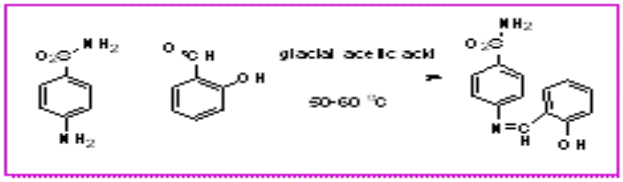

Fig2. Synthesis of Schiff base using glacial acetic acid as a catalyst.

5. Microwave-assisted synthesis of Schiff base

In recent times, the microwave heating technique has developed as an effective technique to stimulate a wide range of chemical reactions. It was demonstrated that the reaction of aniline derivative with various aromatic aldehydes can be performed quickly, with good purity, and without using solvent, by utilizing microwave irradiation aid. The recrystallization method was used to purify the products in several suitable solvents, and showed an excellent yield with high purity [23-27]. Different types of Schiff base compounds were synthesized using microwave technique very quickly; only one or two minutes for a reaction that requires two hours without a microwave, and at room temperature, Figure 3 [28].

6. Schiff base synthesis using natural catalysts.

Utilizing lemon juice as an acid catalyst to prepare Schiff base by the reacting of an aromatic amine with aromatic aldehyde under free solvent conditions demonstrated

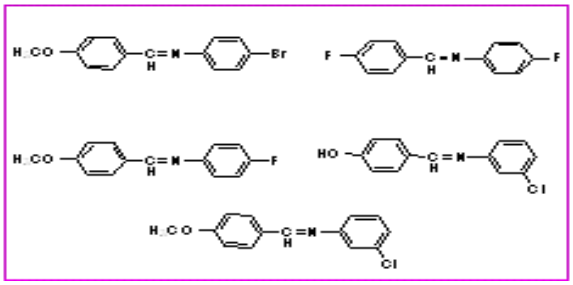

Fig 3. Some chemical structures of Schiff base compounds.

a very good yield, as shown in Figure 4. Using this type of catalysts shows quite essential advantages for the environment, especially when the reaction is solvent-free conditions because almost all of organic solvent is flammable and toxic. It has also exhibited economic reaction, simple workup, and high percentage yield. These solvent-free reactions typically require shorter reaction times as the reaction happens only by mixing the primary amine with aldehyde or ketone at ambient temperature in the presence of lemon as a catalyst. Then the mixture was purified to obtain a yellow crystalline target product with an excellent percentage yield of 94% [29-31].

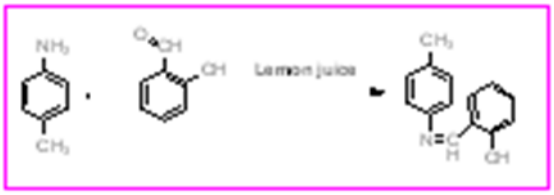

Fig 4. Synthesis of Schiff base using natural acid (lemon juice) catalyst.

7. Synthesis in the presence of UV Rays.

An equimolar amount of p-toluidine and vanillin were mixed and placed in UV Chamber for 15 min, as shown in Figure 5. , The formation of a pale yellow colored product indicates completion of the reaction. The shock cooling recrystallization approach was used to purify the crude product, and it was obtained an excellent yield of about 97% as a nice crystal. Hence in the classical procedure, it gives only 78%. Also, the time required in the traditional method is more, i.e., 1-1.5 hours, whereas using UV light, the reaction time decreased to about 15 minutes [32-35].

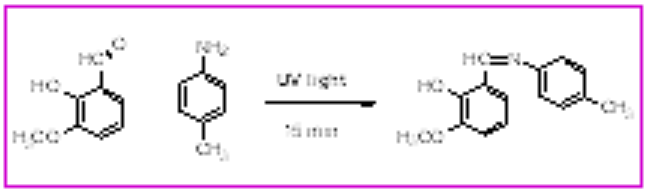

Fig 5. Synthesis of Schiff base compound using UV light.

8. Synthesis by using a sonicator

8.1. Without catalyst.

It was dissolved vanillin (0.05 mole) in methanol (5 mL), and in another container, it was dissolved 0.05 mole of p-toluidine in the same amount of solvent. After that, the two contents were mixed in a beaker, which was placed in a sonicator for about 15 minutes at 44 ºC. A yellow color was observed, which shows the creation of the target compound. The shock cooling recrystallization approach was used to purify the crude product, and it was obtained an excellent yield of about 97% as a nice crystal. Hence the classical procedure gives only 78%, as shown in Figure 6 [36-39].

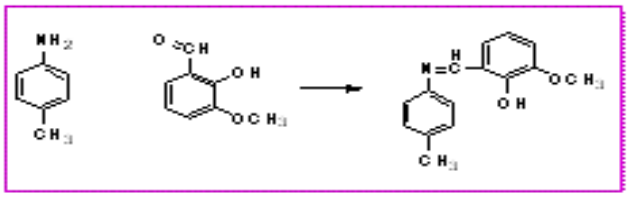

Fig 6. Synthesis of Schiff base molecule without catalyst.

8.2. With catalyst.

0.1 Mole of p-toluidine was added to methanol (5 mL) and stirred until fully dissolved, and 0.1 moles of vanillin was added to methanol (5 mL) in another beaker and stirred until fully dissolved. After that, the contents in both beakers were mixed and a few drops of acetic acid was added as a catalyst, and the beaker was placed in a sonicator at 45 ºC for 9-10 min. A pale-yellow colored product was formed, which indicated the formation of the product. The synthesized product was recrystallized *via* a shock cooling process, utilizing ethanol as a solvent to give fine crystals of the target compound. Compared to the previous method of synthesis by the use of a sonicator without a catalyst, this method has more yield with the use of catalysts. The yield of the Schiff base produced was found to be 98.30%, as shown in Figure 7 [40-42].

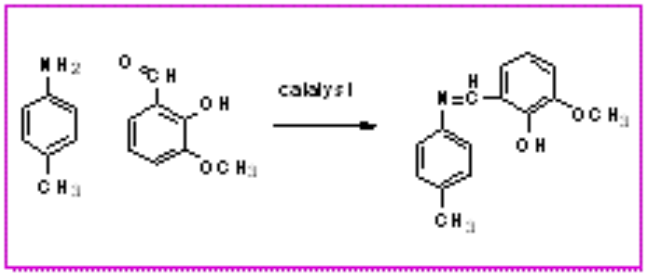

Fig 7. Synthesis of Schiff base molecule with catalyst.

9. By using mortar and pastel

An equimolar amount of p-toluidine and vanillin were put in mortar for about 11 minutes, an alternation in the color of the reaction mixture was noticed. After that, it was kept in a dark place for 24 hours, resulting in a yellow compound, which indicated the formation of the product. The reaction progress was followed by TLC and infrared spectroscopy. The shock cooling recrystallization approach was used to purify the crude product and it was obtained a pure target compound with an excellent yield of about 96% as nice crystals. Linked to the traditional methods, these approaches are more suitable with a higher yield, shorter reaction time, and milder conditions, without making too much of a by-product [43].

10. Reaction mechanism of Schiff base formation

The electron-rich nitrogen atom in the amine group is a nucleophile that attacks the carbon atom, as an electrophile, aldehyde, or ketone group. The outcome of this reaction is the formation of imine or Schiff base functional group C=N instead of carbonyl group C=O of aldehyde or ketone group. Mechanistically, the formation of a Schiff base or imine functional group includes two stages. The first step is the nitrogen atom of the amino group acts as a nucleophile, then attacking the carbon atom of the carbonyl group. This is closely analogous to hemiacetal and hemiketal formation, as shown in Figure 8.

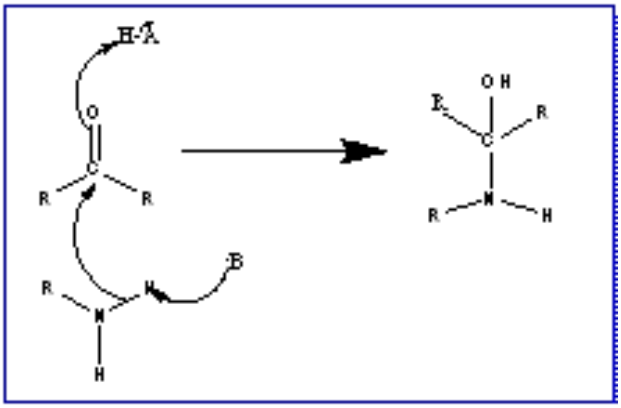

Fig 8. First step of Schiff base mechanism reaction.

In this step, the nitrogen atom of amine was deprotonated using a base, and the nitrogen–hydrogen electrons have pushed the oxygen atom off of the carbon, forming a C=N (Schiff base functional group) and releasing water molecule as shown in Figure 9 [44].

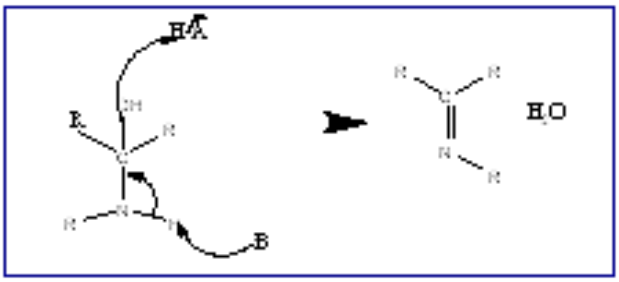

Fig 9. Second step of Schiff base mechanism reaction.

The reversibility of the Schiff base reaction is quite possible by getting back the reactants, which are the primary amine and aldehyde or ketones, as shown in Figure 10 [45-46].

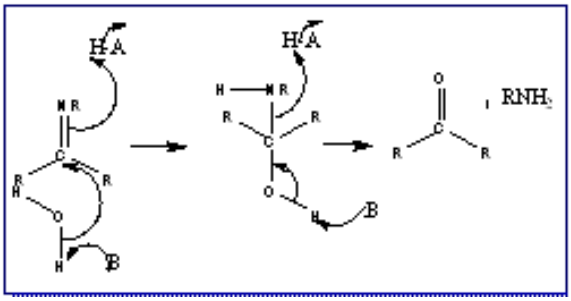

Fig 10. The reversibility of Schiff base reaction.

11. CONCLUSION

In summary, Schiff's base molecules can be prepared by reaction of primary amines with aldehyde or ketone with realizing H_2O molecule and forming imine functional group –C=N–. The scientist Hugo Schiff who first synthesized imine functional group, and it is named according to his name. Because of their interesting biological activity, the Schiff's base compounds have gained special attention. Chemists have synthesized thousands of compounds as antibiotics and anti-inflammatory, which contains imine groups. In addition, Schiff's base molecules have also been used in industries such as plastic stabilizers, inhibitors of metallic corrosion, and dyes. In this work, it is an attempt to focus on the recent methods of imine group preparation and their applications. The mechanism of the reaction was also highlighted and also explained why the reaction is reversible.

12. Funding

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13. Conflicts of Interest

The authors declare no conflict of interest.

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نظرة عامة: استخدام مختلف الطرق لتحضير قواعد شيف الجديدة

اناهيد ياسين[1]، عماد التكريتي[1]، محمد المشهداني[2]، نادية صالح[3]، عماد يوسف[2]

[1] قسم الكيمياء ، كلية العلوم ، جامعة تكريت ، تكريت ، العراق [2] قسم الكيمياء ، كلية العلوم ، جامعة النهرين ، بغداد ، العراق

[3] قسم العلوم الكيميائية ، كلية العلوم والتكنولوجيا ، الجامعة الوطنية الماليزية ، بانجي ، سيلانجور ، ماليزيا

emad_yousif@hotmail.com

الخلاصة:

سميت مركبات قاعدة شيف نسبة إلى العالم الذي حضرها لاول مرةً (هوغو شيف). يتم تصنيعها عن طريق تفاعل التكثيف لمجموعة الكربونيل–C=O– لمركب الألدهيد أو الكيتون مع أمين أولي. وهذا يؤدي إلى تكوين مجموعة الإيمين–C=N–بالإضافة إلى جزيء الماء. اكتسبت جزيئات قاعدة شيف أهمية خاصة بسبب نشاطها البيولوجي، مثل مضادات الالتهاب والمضادات الحيوية. تُستخدم مركبات قاعدة شيف أيضًا في الصناعة كمثبطات للتآكل، وأصباغ، ومثبتات ضوئية للبوليمرات البلاستيكية. تتضمن هذه المراجعة القصيرة تسليط الضوء على المناهج الحديثة لتركيب جزيئات قاعدة شيف الجديدة. ويناقش أيضًا آلية التفاعل ولماذا يكون تفاعل التكثيف قابل للانعكاس.

الكلمات المفتاحية: *قاعدة شيف، آزوميثين، مجموعة الامين، تفاعل النيوكليوفيلي، تفاعل التكثيف.*