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(RESEARCH ARTICLE)

SYNTHESIS OF SCHIFF BASES FROM TRIAZINES

Heshw nozad yousif *, Bekhal Omer Mohammed and Muthanna M. AWAD

Department of Chemistry, College of Science, University of Kirkuk, Kirkuk, Iraq.
* Corresponding Author

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ABSTRACT

This review summarizes the chemistry, synthesis, applications, and characterization of triazine-derived Schiff bases, with emphasis on the integration of the 1,3,5-triazine core with azomethine (C=N) functionalities. The principal synthetic strategies include the use of amino-functionalized triazines such as melamine, aldehyde-functionalized triazine cores, and imine-linked covalent organic frameworks. Biological and pharmaceutical activity, heavy-metal sensing, corrosion-related surface interactions, fluorescence, bioimaging, and optoelectronic applications are discussed in relation to the structural features of the triazine and Schiff-base units. The review also highlights advanced synthetic approaches, including microwave-assisted, one-pot, and solvothermal methods, which can reduce reaction time and solvent consumption while enabling access to structurally complex products and crystalline frameworks. Finally, the main spectroscopic and physical characterization methods used for structural confirmation are outlined, including FTIR, NMR, elemental analysis, mass spectrometry, TGA, and DSC. Overall, triazine-derived Schiff bases represent versatile molecular and framework systems with broad potential in coordination chemistry, sensing, biological applications, and functional materials.

Keywords: Triazine – derived Schiff bases; Azomethine (C=N) chemistry; Covalent organic frameworks; Heavy – metal sensing; Microwave – assisted synthesis; Spectroscopic characterization.

1 INTRODUCTION AND LITERATURE REVIEW

1.1 Schiff Bases

Schiff bases are a very important class of compounds in organic and coordination chemistry. They are aldehyde or ketone derivatives where the carbonyl group (C=O) has been replaced by an imine or azomethine group (C=N) via condensation with primary amines [1]. The azomethine functionality is of special importance as the nitrogen atom has a lone pair of electrons which makes Schiff bases excellent ligands and the stability of the complexes with transition-metal ions is remarkably high [2]. Apart from their application in coordination chemistry, recent reports indicate that a variety of biological activities have been associated with Schiff bases. The azomethine group is often considered to be a vital pharmacophoric element responsible for antibacterial, antioxidant, and anticancer properties especially within heterocyclic moieties [3].

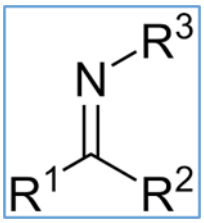


Figure 1: General structural representation of a Schiff base containing the azomethine (C=N) linkage.

1.2 Chemistry of 1,3,5-Triazine

Triazine rings are a significant type of six-membered aromatic heterocycle. Among the triazine isomers, 1,3,5-triazine (s-triazine) is the most stable and it is considered as the most important one in industrial and pharmaceutical areas. Due to high electron negativity of three ring nitrogen atoms, carbon atoms in triazine ring becomes electron deficient and this property of triazine makes it prone nucleophilic aromatic substitution (SNAr) reactions [4].

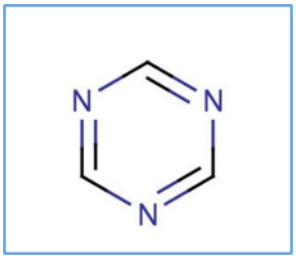


Figure 2: Chemical structure of the 1,3,5-triazine (s-triazine) ring.

Cyanuric chloride is also an important starting material for the preparation of triazine derivatives, since its three chlorine atoms can be substituted stepwise under thermo-regulated conditions. This allows for the synthesis of star-shaped macromolecules, or covalent polymeric networks, e.g. covariant organic frameworks (COFs). Employing the triazine nucleus as a focal core to which other substituents can be tethered confers structural rigidity and enhanced thermal stability to the resultant molecules [4,5].

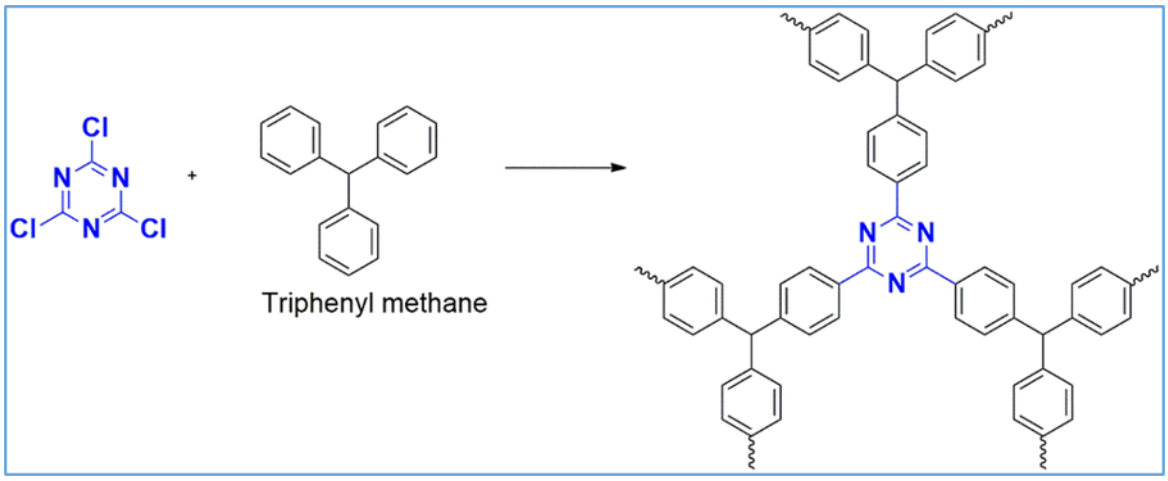


Figure 3: Representative functionalization of the triazine core through substitution reactions of cyanuric chloride.

1.3 Strategies for Integrating Triazines with Schiff Bases

Because of the considerable structural diversity offered by triazine chemistry, synthetic strategies for triazine-derived Schiff bases can be divided into two principal routes reported in the literature. Triazine rings have attracted substantial attention in organic synthesis, and several approaches have been developed to combine them with Schiff-base functionalities. These approaches may be classified according to the role of the triazine unit in the reaction: either as a source of amino groups or as a source of aldehyde groups.

1.3.1 Triazine as an Amine Core

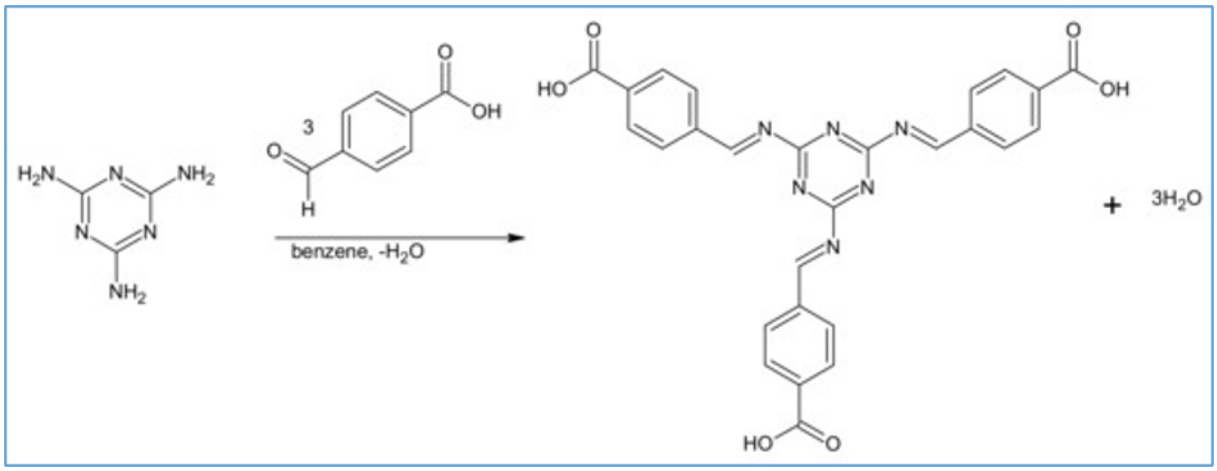


Figure 4: Representative synthesis of a melamine-derived tripodal Schiff base by condensation with aromatic aldehydes.

In this route, triazine derivatives bearing free amino groups are used, most notably melamine (2,4,6-triamino-1,3,5-triazine). The three amino groups can react with aromatic or aliphatic aldehydes to form three-armed Schiff bases. Such compounds are characterized by high molecular symmetry and by extensive intermolecular hydrogen-bonding networks [6]. Melamine is one of the best-known triazine derivatives containing three primary amino groups [10]. In a study reported in 2018, a series of tripodal Schiff bases was prepared by direct condensation of melamine with different aromatic aldehydes, including benzaldehyde and halogen-substituted benzaldehyde derivatives. The reactions were carried out in ethanol using a few drops of glacial acetic acid as a catalyst under reflux for several hours [11]. The resulting compounds were reported to exhibit high thermal stability together with strong intermolecular association.

1.3.2 Triazine as an Aldehyde Core

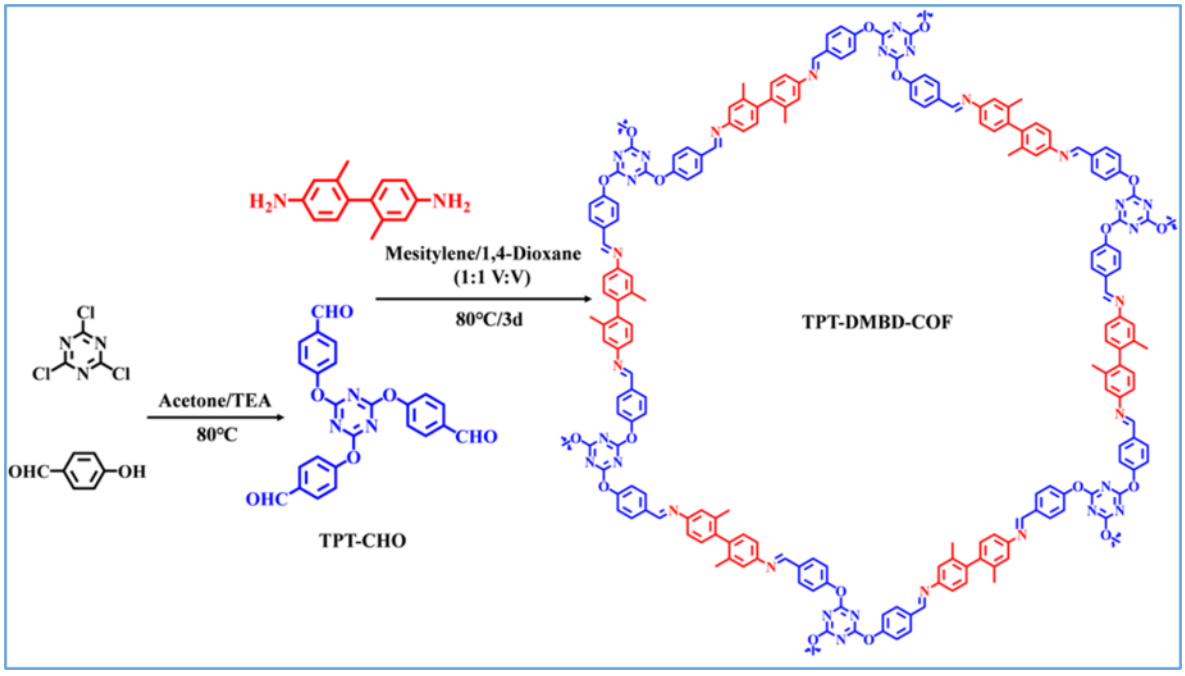


Figure 5: Representative construction of a triazine-based imine-linked framework from an aldehyde-functionalized triazine precursor.

This route is comparatively more complex and recent. It commonly begins with cyanuric chloride, followed by nucleophilic substitution reactions that introduce aldehyde-bearing groups onto the triazine ring, for example through reaction with 4-hydroxybenzaldehyde. The resulting aldehyde-functionalized triazine is then reacted with various amines to form Schiff bases. This strategy provides greater control over the steric and electronic properties of the final product [7]. One representative synthesis: 2,4,6-tris(4-formylphenoxy)-1,3,5-triazine was prepared by a nucleophilic substitution reaction of 4-hydroxybenzaldehyde with cyanuric chloride under basic conditions in [7]. The aldehyde-

terminated core can then be reacted with various aromatic amines, including aniline derivatives, to afford star-shaped Schiff bases. These reactions are ordinarily carried out in polar aprotic solvents like DMF or DMSO to enhance the solubility of the hefty molecular materials [6].

1.3.3 Triazine-Schiff Bases in Covalent Organic Frameworks (COFs)

Covalent organic frameworks (COFs) are arguably the most talen applications of SchiffBASE chemistry in the materials science field. Recently, triazine-based imine condensation has adapated towards the synthesis of porous two- and three-dimensional polymer networks by combination of multifunctional amino-triazine monomers with multi-aldehyde monomers [8]. The commercially available azomethine, in a robust two-component system joined by the azomethine (C=N) linkage, displays exceptional porosity and chemical stability, has been proposed for gas storage, hydrogen and carbon dioxide, and molecular separations and water treatment [9]. The molecular design of the synthesized cofs are shown in Figure 6 . The model is a regular hexagonal network, with well-defined pores acting as adsorption sites for gases or pollutants. The side view also illustrates the mechanism for our framework to form ordered two-dimensional layers, which stack with a geometrically regular manner, through strong covalent imine linkages that connect the triazine to a rigid network [8].

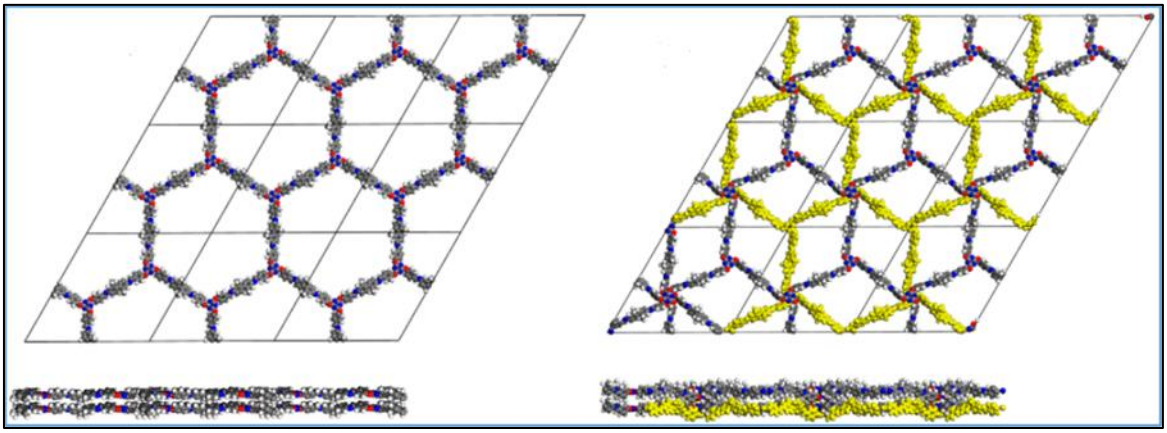


Figure 6: Molecular model illustrating the porous architecture of covalent organic frameworks (COFs) and their layered stacking pattern.

1.4 Applications of Triazine-Derived Schiff Bases

The rigidity of the triazine ring and the high coordination ability of the azomethine (C=N) group endowing the Schiff-base systems with numerous application possibilities. Main topics in the literature are briefly outlined below.

1.4.1 Biological and Pharmaceutical Applications

Triazine-based Schiff bases have been reported in the literature to inhibit a wide range of bacteria and fungi [15-20]. This effect was attributed to the interaction of the imine group with biological target and the well-known relevance of triazine scaffold in numerous biologically active and pharmaceutical agents [10].

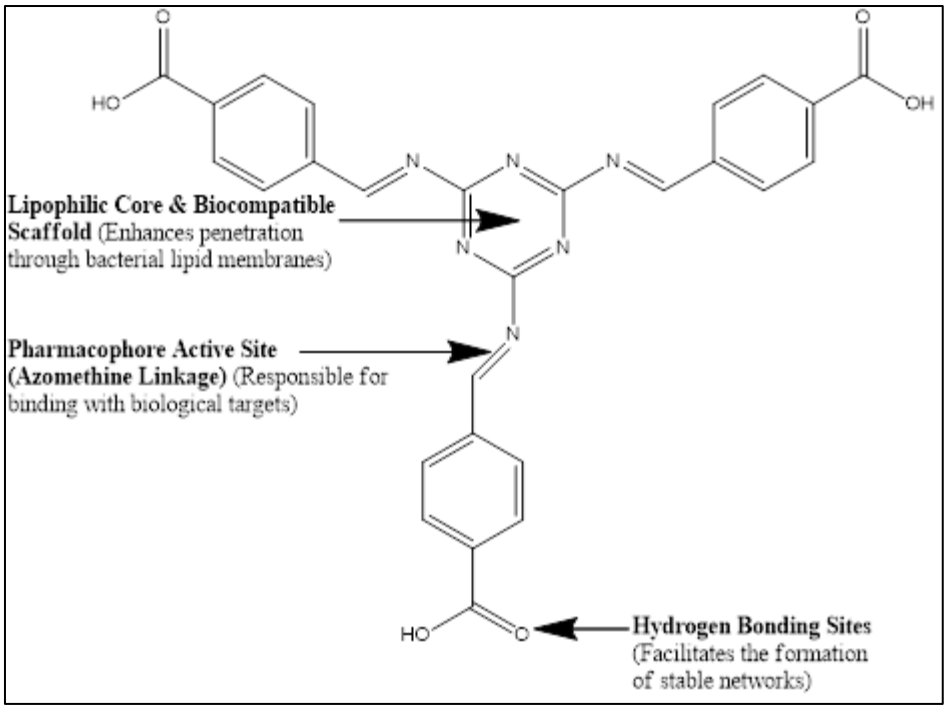


Figure 7: Structural features associated with the proposed structure–activity relationship of the star-shaped triazine-derived Schiff base.

1.4.1.1 Structure-Activity Relationship (SAR)

The above scheme represents the prepared star-shaped compound's structure and the activity relationship and the structural key point which is supposed to lead to its bio and pharmaceutical activity. These attributes can be classified into three main elements.

1.4.1.1.1 Triazine Core: Lipophilic Core and Biocompatible Scaffold

The central triazine ring acts as the core of the star-shaped molecule. Its lipophilic nature and the property of being a scaffold have been related to interactions with lipid membranes [11] and it has been suggested that they might be able to cross into bac teria and fungal cells.

1.4.1.1.2 Azomethine/Imine Linkage: Pharmacophoric Active Site

The C=N groups in the three arms of the molecule are the main pharmacophoric centers. Such moieties may directly interact with biological target (ie, proteins and enzymes related to microbial cell) and may lead to microbial growth inhibition [12].

1.4.1.1.3 Terminal Carboxyl Groups: Hydrogen-Bonding Sites

Those compounds have high symmetry and contain terminal carboxyl group(–COOH) which can generate strong intermolecular hydrogen bonding. Such interactions aid to the establishment of robust supramolecular networks and may enhance the compound's affinity for biological receptors in target cells [13].

1.4.2 Colorimetric and Fluorescent Chemosensors for Heavy-Metal Ions

As these molecules have multiple nitrogen donors, i.e., the nitrogen located in the triazine ring as well as in the imine groups (-CH=N-), they can provide good coordination with metal ions. These sites of coordination may bind with toxic and heavy Metal like Co2+ and Hg2+ selectively. The compounds can act as sensitive chemosensors for environmental contaminants in water because metal complexation may result in color or fluorescence changes [14,15].

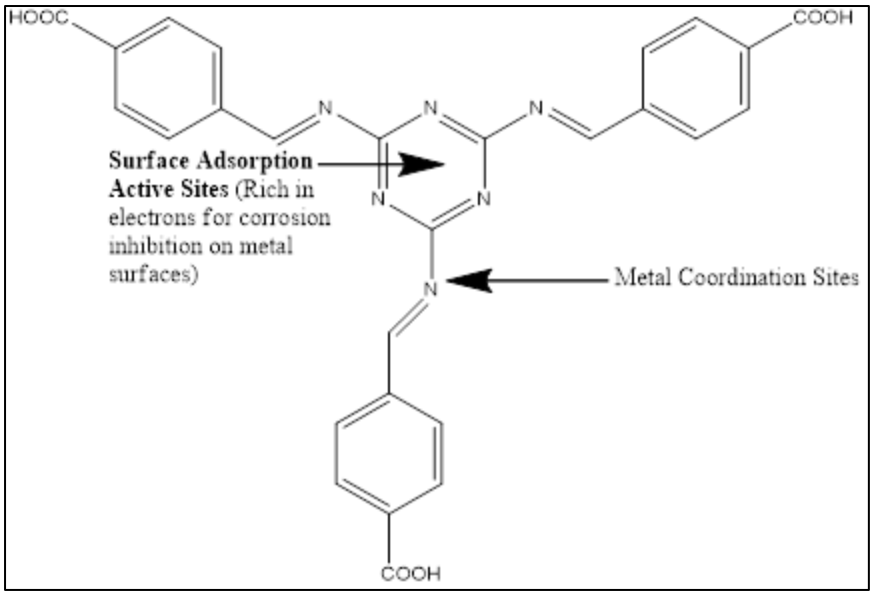


Figure 8: Metal-coordination and surface-adsorption sites of a triazine-derived Schiff base relevant to sensing and corrosion-inhibition applications.

The above diagram also summarizes the key industrial and physicochemical applications that can be considered through metal-coordination sites and active surface-adsorbate sites.

1.4.2.1 Metal-Coordination Sites

The consecutive nitrogen atoms of the azomethine linkage possess lone electron pairs and thus offer appropriate coordination sites for heavy-metal ions like Co2+ and Hg2+. Complexation may trigger a colorimetric or fluorometric response, which allows the compound to be used as a chemical sensor for metal-ion contamination [15].

1.4.2.2 Active Surface-Adsorption Sites

The aromatic rings and delocalized pi-electron systems may induce physical as well as chemical adsorption on metal surfaces like steel. Therefore, the adsorbed species could produce a thin film on the metal surface, which reduces the contact between the metal and acidic solution so that the compound can be considered as a corrosion inhibitor [16].

1.4.3 Photophysical Properties and Fluorescence Applications

Star-shaped 1,3,5-triazine-based compounds may exhibit unique photophysical properties due to their large π-conjugated systems. The central triazine moiety can also be considered as an electron acceptor, with conjugation being extended through the schiff base azomethine linkages (C=N) towards the peripheral aromatic moieties. Such extended conjugation facilitates electronic transitions such as pi-pi* transitions and in some systems allows absorption of energy from the sun in the ultraviolet region followed by emission in the visible region as fluorescence [17,18]. These architectural features enable a variety of functional applications, including bioimaging and optoelectronics.

1.4.3.1 Bioimaging

The fluorescence of Schiff-base derivatives can be utilized by employing these compounds as fluorescent probes for biological applications. Since the Schiff-base bond is sensitive to its local environment and can bind to biological cells such as bacterial and cancer cells, it is possible to visualize the labeled systems and track the fluorescent probe distribution with high contrast using fluorescence microscopy [17,19].

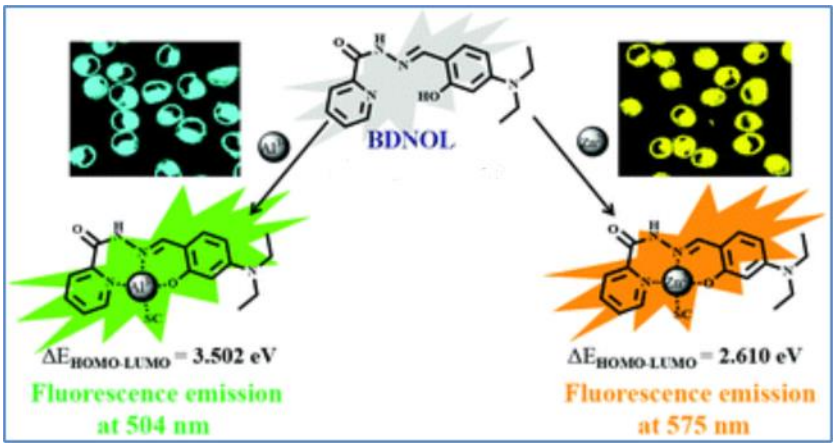


Figure 9: Fluorescence mechanism.

Fluorescence emission in this class of compounds is associated with an extended conjugated electronic system involving the triazine core and Schiff-base C=N linkages. In metal-responsive systems, enhancement of fluorescence can be interpreted through the chelation-enhanced fluorescence (CHEF) mechanism [17,20], as outlined below.

Free state (before binding). The compound may exhibit weak fluorescence emission because rotational freedom around the imine-containing framework provides nonradiative pathways through which absorbed energy is dissipated as molecular motion and thermal vibration rather than being released as light [17].

When the Schiff-base nitrogen atoms coordinate to the target metal ions, the molecular rotation is more hindered and the structural rigidity is enhanced. This constriction minimizes nonradiative energy dissipation and enhances fluorescence emission. This 2 signaling pathway can generate a turn-on sensing effect in which the fluorescence level is significantly enhanced after target binding, resulting in high-contrast signals for sensing and bioimaging applications [20,21].

1.4.3.2 Advanced Optoelectronic Applications (OLEDs).

Owing to the high thermal stability of the triazine core and its electron-transporting ability, star-shaped triazine systems are considered promising materials for organic light-emitting diodes (OLEDs), modern displays, and high-efficiency optoelectronic sensors [22,23].

Figure 1-7. Schematic illustration of a triazine-centered star-shaped architecture and its potential optoelectronic applications.

1.4.3.2.1 1,3,5-Triazine Core

The triazine unit forms the central part of the molecule and contributes to electron withdrawal and transport within optoelectronic devices [24].

1.4.3.2.2 Conjugated Arms

The conjugated peripheral arms are responsible for much of the luminescent behavior and can contribute to tunable emission color [25].

1.4.3.2.3 High Thermal Stability

The Star-shaped architecture Endows Strong thermal stress resistance, which is a vital factor to sustain stability and longevity of display and optoelectronic materials [23].

1.4.3.2.4 OLED Display Applications

The intense emission and good electron-transport properties of these compounds suggest that they could be attractive candidates for high-performance contemporary and bendable organic screens [23, 24].

1.4.3.2.5 Optoelectronic Sensors

Such architectures find applications even in adaptive sensing systems, including optical and other electroluminescent platforms for sensing [25].

1.4.3.2.6 Cell Imaging

Besides their potential in electronics, the conjugated triazine systems can serve as fluorescent probes for live cell imaging in fluorescence microscopy [25].

2 ADVANCED SYNTHETIC METHODOLOGIES FOR TRIAZINE-BASED SCHIFF BASES

Modern synthesis endeavors targeting triazine-based Schiff bases have [26,27] increasingly diverged from the classical reflux methods which long reaction times and organic solvents were often mandatory, to more efficient procedures, greener reaction media and advanced building block strategies. The major synthetic routes found in the literature are outlined below.

2.1 Microwave-Assisted Synthesis

MW/US – Microwave irradiation/ultrasound MWI – Microwave irradiation MWT – Microwave treatment 1.2 Synthesis of Schiff base Triazines The MWI is a superior contemporary method for the fast production of triazine derivatives, including related Schiff-base compounds. Recently a study was published in ACS Omega to compare the conventional and microwave methods in synthesis of triazine derivatives. Microwave was applied on traditional synthesis of α-aminonitriles and significantly reduced reaction time from several hours to minutes under conventional reflux method, meanwhile the yields have been enhanced, and the by-products were decreased. The method exploits the sudden, relatively even, increase in tem- perature in the reaction medium which speeds-up reaction kinetics and may help in condensation reactions including water elimination and formation of azomethine linkage [28].

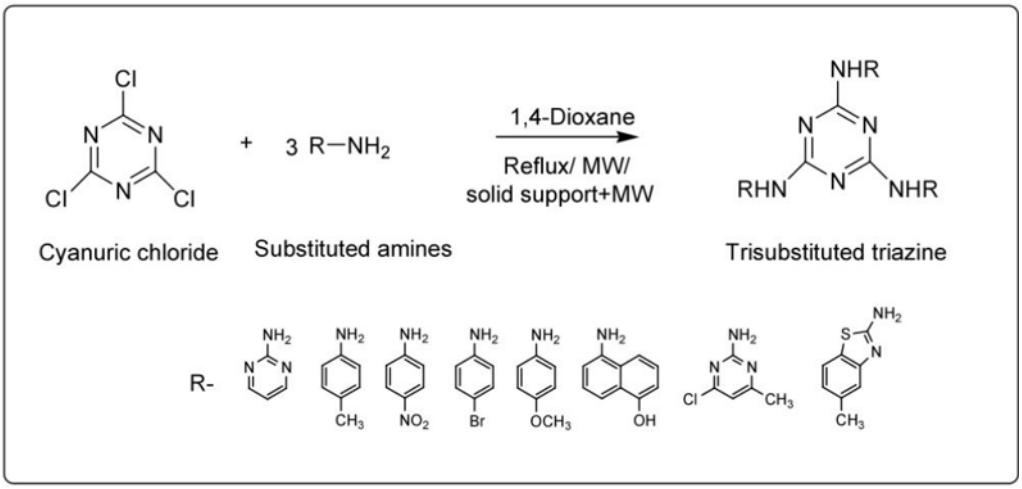


Figure 10: General synthesis of trisubstituted 1,3,5-triazine derivatives under conventional or microwave-assisted conditions.

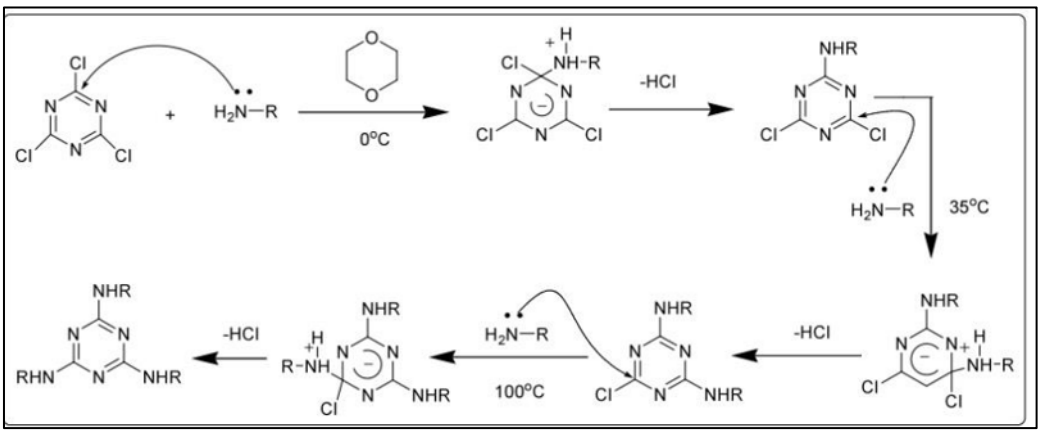
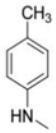
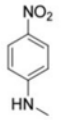
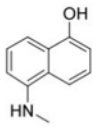
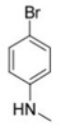
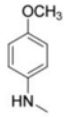
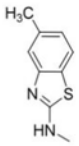
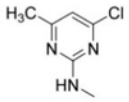


Figure 11: Stepwise nucleophilic substitution of cyanuric chloride with amines at controlled temperatures.

Table 1: Comparison of conventional and microwave-assisted methods for the synthesis of triazine derivatives.

Compd.	Substituent R	Conventional		MW-Assisted		MW-Assisted Solid-supported			
						Organic support		Inorganic support	
		Reaction time	% yield	Reaction time	% yield	Reaction time	% yield	Reaction time	% yield
S1		6 h	76%	2 min 50 sec	87%	1 min 5 sec	93%	1 min 40 sec	90%
S2		6 h	78%	2 min 55 sec	86%	55 sec	94%	1 min 10 sec	90%
S3		6 h	77%	2 min 40 sec	86%	50 sec	93%	1 min 15 sec	89%
S4		6 h	74%	3 min	85%	50 sec	92%	1 min 10 sec	88%
S5		6 h	73%	2 min 40 sec	84%	60 sec	92%	1 min 20 sec	87%
S6		6 h	76%	2 min 30 sec	86%	1 min 10 sec	95%	1 min 35 sec	92%
S7		6 h	68%	2 min 55 sec	79%	1 min 15 sec	94%	1 min 40 sec	88%
S8		6 h	66%	2 min 40 sec	79%	60 sec	91%	1 min 20 sec	86%

2.2 One-Pot Synthesis

One-pot methods are prevalent in contemporary organic synthesis as starting materials, for example aldehydes and amino-functionalized triazine derivatives, may be introduced along with the necessary reagents or catalysts into one reaction vessel without the need to isolate or purify labile intermediates. This approach was recently also extended for the preparation of elaborate hybrid triazine derivatives, where an in situ Schiff-base intermediate is generated and then undergoes rearrangement or cyclization within the same pot. This method may minimize material loss and significantly cut down on solvent use[29].

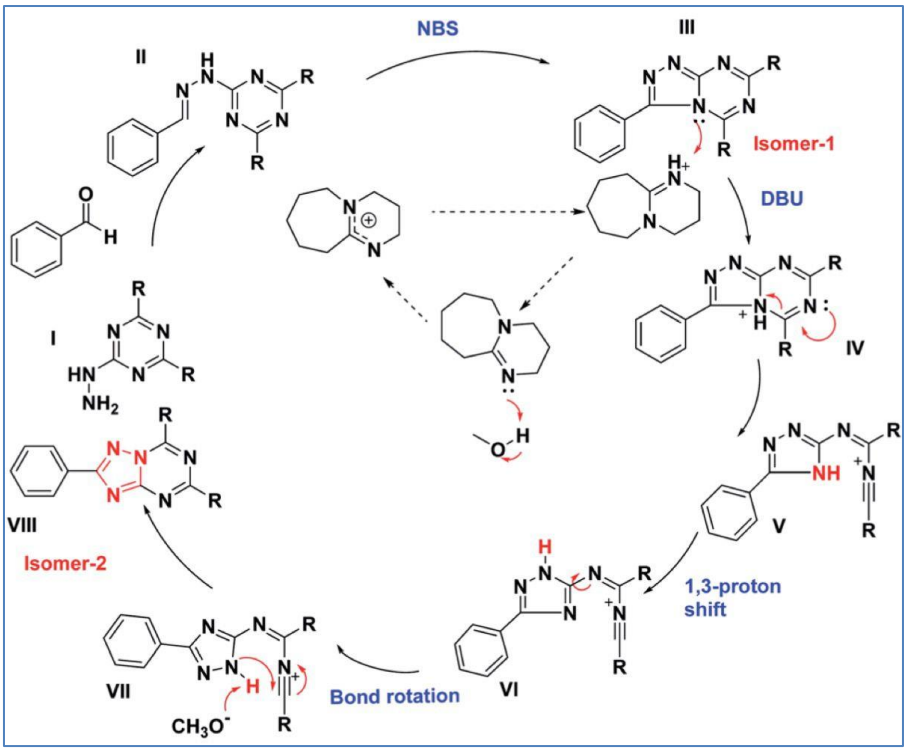


Figure 12: Proposed mechanism of the one-pot synthesis, showing formation of a Schiff-base reactive intermediate (compound II) followed by a DBU-mediated Dimroth rearrangement.

2.3 Solvothermal Synthesis for the Construction of Crystalline Frameworks

Solvothermal methods have been exploited to produce covalent organic frameworks (COFs) with triazine and Schiff-base moieties. In this method, a multifunctional triazine monomer and the complementary reactants are combined in a Teflon-lined autoclave at high pressure and temperature, which can be up to ca. 120 °C, for a few days. These mild conditions are sufficient for the reversible condensation to yield highly ordered crystalline porous structures. Reversibility at early times enables a rearrangement of the structure to a spatially more stable one. Such networks have been exploited in gas adsorption and molecular separations. Figure The Teflon-lined stainless-steel autoclave employed in solvothermal synthesis to furnish the autogenous pressure and heat for COF crystal growth [30].

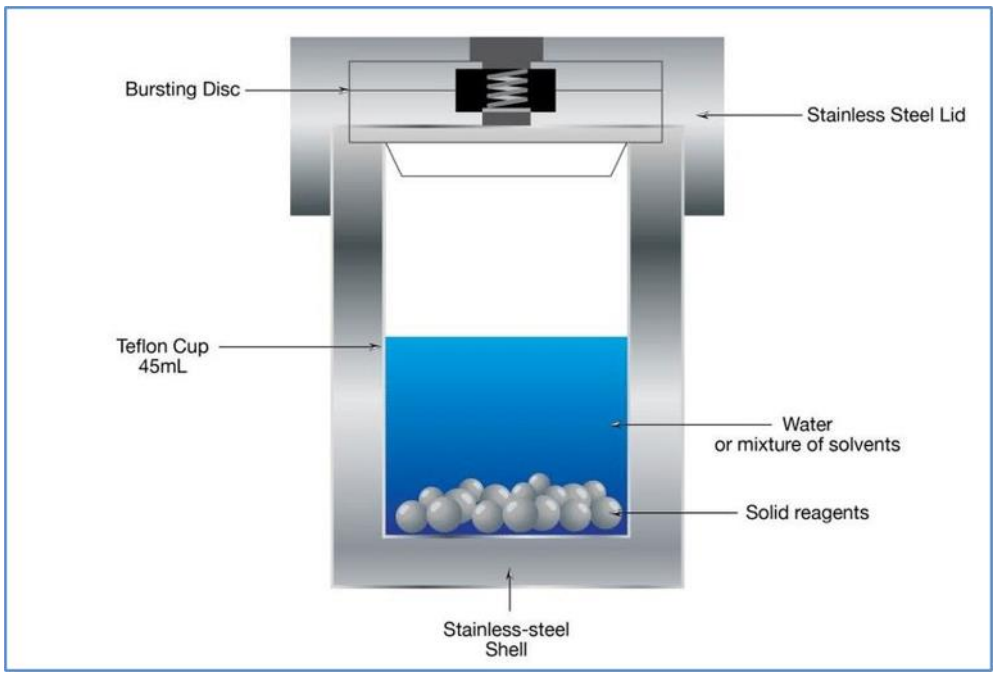


Figure 13: Teflon-lined stainless-steel autoclave used for solvothermal synthesis.

3 SPECTROSCOPIC AND PHYSICAL CHARACTERIZATION OF TRIAZINE-BASED SCHIFF BASES

Following the synthesis of triazine based schiff bases, several analytical methods are routinely used to confirm the chemical structure and the purity of the synthesized materials. The main techniques to describe are the following.

3.1 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy serves as a quick preliminary method for monitoring the success of a condensation reaction and the formation of a Schiff-base linkage. Structural identification is in general supported by the vanishing of typical bands of the substrates and the arising of new bands in the products.

The strong aldehydic carbonyl (C=O) band at ca. 1703 cm^{-1} and the bands belonging to the amino-group (NH2) of the precursor at the 3466-3207 cm-1 region vanish upon condensation.

The resulting covalent framework also exhibits a new sharp absorption band, due to the stretching vibration of the azomethine group (C=N), which appears at around 1618 cm^{-1}.

The central triazine characteristic bands can still be found around 1560cm^{-1}, which means the core remained stable during the reaction. The FTIR spectrum of the synthesized TPT-DMBD-COF triazine based covalent organic framework demonstrates the modification which occurs upon framework formation. The distinctive absorption bands associated with the functional groups of the reactants, especially those of the aldehyde carbonyl (C=O) and amino (NH_2) disappears, indicative of the usage these functional groups in the reaction. However, a sharp new intensity peak is found in the region 1639–1590 cm-1 which is due to a stretching vibration of the newly generated azomethine (C=N) group and this confirms the production of the Schiff-base network. The spectrum is also characterized by distinctive peaks of the central triazine ring, suggesting the existence of the core framework [8].

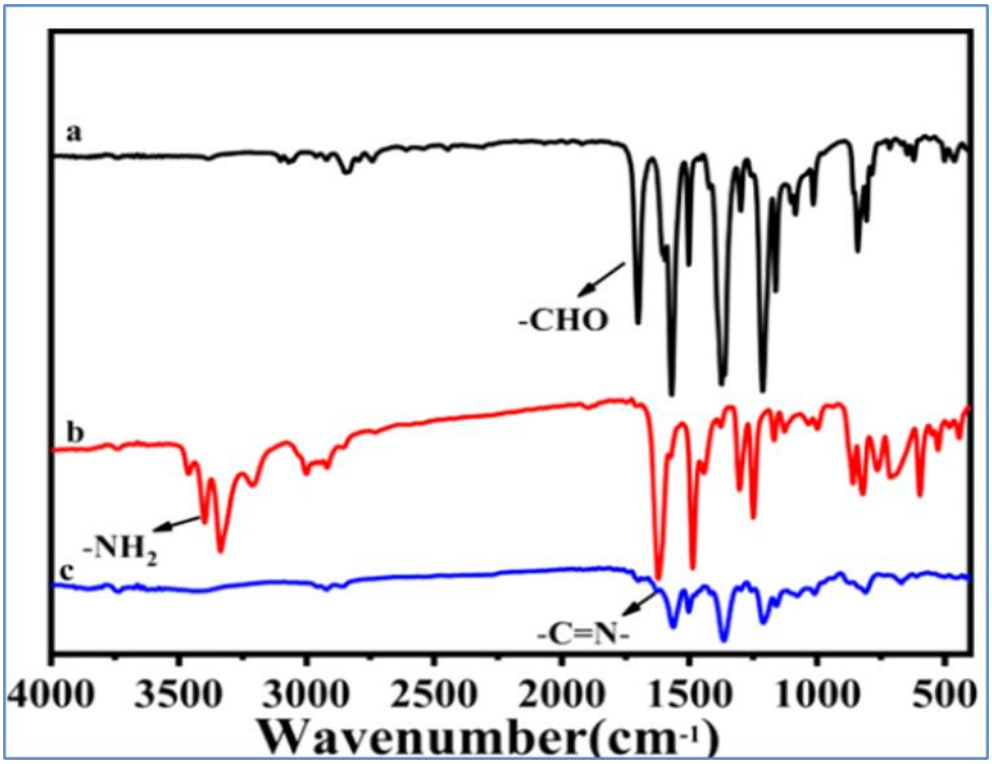


Figure 14: Comparative FTIR spectra of the TPT-DMBD-COF and its starting monomers.

3.2 Nuclear Magnetic Resonance Spectroscopy (1H NMR and 13C NMR)

NMR spectroscopy provides detailed information about the chemical environments of hydrogen and carbon atoms and is an important tool for confirming formation of the covalent linkages in the resulting TPT-DMBD-COF system.

^{1}H NMR. Because covalent organic frameworks are generally insoluble in common solvents, this spectrum is used in the source study to follow the monomer-related signals. The most diagnostically important signal is assigned to the azomethine proton (HC=N). Owing to deshielding by the adjacent nitrogen atom, this proton appears as a singlet in the downfield region at approximately 8.1-8.9 ppm. The proton signal of the aldehyde starting material, usually detected

at >9.5 ppm, vanishes upon condensation. Aromatic protons were observed at 6.5-8.0 ppm with integration values in agreement with corresponding proton environments [31].

Solid-State 13C NMR (CP/MAS). This method confirms the carbon skeleton of the end solid product. The azomethine carbon (HC=N) is found in the 155-165 ppm region. The three equivalent carbon atoms of the central triazine ring also exhibits a unique downfield signal at about 165-171 ppm due to the fact that they are directly attached to highly electronegative nitrogen atoms, which confirmed the preservation of the triazine ring [8].

3.3 Elemental Analysis and Mass Spectrometry

Elemental Analysis (CHN). Elemental analysis is performed by theoretical factor mass percent content of C, H, and N and experimental values were compared. In the source text, the degree of agreement to be expected between these values, as an indication of purity of the sample is given. Mass Spectrometry. To confirm molecular mass, particularly of large molecules or macromolecular products, which arise from binding several arms to a triazine ring. The presence of the molecular-ion peak (M+) at the expected mass serves as further evidence for the assignment of the molecular formula of the synthesized compound [32].

3.4 Thermal Analysis (TGA and DSC)

Since triazine-based Schiff bases are usually regarded as candidates for the material engineering field and covalent organic framework, it is quite essential to analyze their thermal stability. Thermogravimetric Analysis (TGA)TGA is a technique used to assess mass loss as a function of temperature and has been employed to infer the high thermal stability of triazine-based materials. The compounds outlined in the source text are usually only starting to decompose at high temperatures, in many cases ca. 300-400 °C, which makes them candidates for use in high temperature environment [31].

Differential Scanning Calorimetry (DSC). DSC is employed to detect physical thermal transitions, as opposed to mass loss sequences measured by TGA. The melting temperature, where applicable the glass transition temperature, Tg, of a network of polymeric or macromolecular Schiff-base is obtained from solid state Tg determinations. Such properties are necessary to evaluate the processing and service temperature limits of the material [33].

STATEMENTS ON COMPLIANCE WITH ETHICAL STANDARDS

Disclosure of conflict of interest

The authors declare no conflicts of interest regarding this manuscript".

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