

Article

Design and Structural Evaluation of Melamine-Based Porous Schiff Bases for Enhanced H₂ (gas) Storage

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Abstract: High fuel consumption due to high energy demand has resulted in the use of hydrogen gas as a fuel H₂ (gas) can be stored physically as either a liquid or a gas. In order to store H₂ (gas) as a gas, high-pressure tanks are frequently required (tank pressure of 350–700 bar, or 5,000–10,000 psi). With a boiling point of 252.8 °C at one atmosphere of pressure, H₂ (gas) requires cryogenic temperatures to be stored as a liquid. H₂ can also be retained in solids or on their surfaces (via adsorption/absorption). H₂ (gas) storage is a key enabling technology for the development of H₂ (gas) and fuel cell technologies in industries like fixed power, portable power, and mobility. The development of novel storage technologies with the potential for better energy density is required since H₂ (gas), despite having the highest energy density per mass of any fuel, has the lowest energy density per unit volume due to its low ambient temperature density. The absence of practical, affordable, and secure H₂ storage solutions restricts the use of H₂ (gas) as a fuel. The Schiff bases showed a tiny Brunauer-Emmett-Teller surface area (4.9–10.8 m²/g), typical pore diameters of (1.67 to 3.353 nm), and pore volumes ranging from 0.005 to 0.036 cm³/g. At 77 K and 40 bars, the Schiff bases demonstrated impressive H₂ uptake (0.050377mmol/g), volume of H₂ uptake by Absorbed (1.129143cm³/g,STP) the high uptake appear in Schiff base with the 4-nitrophenyl substituent but Schiff base with the hydroxyl substituent appear H₂ uptake (0.002775mmol/g), volume of H₂ uptake by Absorbed (0.062193 cm³/g,STP). Because it was the most effective medium for H₂ adsorption, the Schiff base with the 4-nitrophenyl substituent can be employed as a gas sorbent.

Keywords: Melamine Schiff bases; porous materials; hydrogen storage; hydrogen adsorption; porous organic polymers.

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1. Introduction

The absence of infrastructure prevents H₂ (gas) from being used as a fuel widely. developing practical, affordable, and secure H₂ storage techniques [1,2]. Department of Energy (DOE) Targets for Onboard H₂ Storage in Automotive Applications cars haven't altered all that much during the previous 13 years [3]. 40 g H₂/L and 5.5%, respectively, were established for five last year. Due to the fact that temperature and pressure are taken into account when determining H₂ storage capacity, these objectives are particularly challenging.

The hydrogen (H₂) storage capacity of materials generally rises under higher pressures but diminishes with increasing temperature. Nevertheless, employing high-pressure systems (requiring specialized containment) or cryogenic cooling (with associated techniques) often results in greater cost and weight, thereby lowering overall efficiency. Research has explored zeolites combined with metal hydrides [4-6], metal-organic

activated carbon [7], with extensive investigations devoted to carbon nanotubes [8], metal–organic frameworks (MOFs) [9], and porous polymers [10]. Because of their broad applications in heterogeneous catalysis [11], gas separation [12], organic vapor sorption, and gas storage, porous organic polymers have become a particularly dynamic research field in recent years [13,14]. These polymers can be synthesized and tailored through diverse organic reactions, are typically free of heavy metals, stable in ambient air, easy to process, mechanically robust, and composed of light elements such as C, H, N, and O [15]. For H₂ storage applications, Covalent organic frameworks (COFs), polymers with intrinsic microporosity (PIMs), and hypercrosslinked polymers (HCPs) are a few examples of porous organic polymers that have been developed [16]. For hydrogen storage, several classes of porous organic polymers have been introduced, including covalent organic frameworks (COFs), polymers of intrinsic microporosity (PIMs), and hypercrosslinked polymers (HCPs) [17]. COFs, which consist of covalently bonded organic units forming crystalline porous networks, can store up to 1.699 wt% H₂ at 77 K and 1 bar [18]. PIMs, characterized by rigid molecular backbones that hinder efficient packing, have demonstrated adsorption capacities of 2.7 wt% H₂ at 77 K and 10 bar [19–22]. HCPs, on the other hand, exhibit large surface areas, permanent porosity due to extensive crosslinking, and hydrogen storage capacities reaching 3.7 wt% [23–26].

Although multiple synthetic strategies exist, most rely on costly precursors, noble metal catalysts, or strong Lewis acids [27], making low-cost and straightforward production challenging.

Melamine (MA) is an inexpensive triazine-based compound widely used in paper, plastics, and pharmaceuticals. It has been employed to synthesize nitrogen-rich porous frameworks suitable for various applications, including hydrogen storage [28–30]. Recently, Schwab and colleagues developed a distinctive one-step condensation method combining MA with aromatic aldehydes in dimethyl sulfoxide via Schiff base chemistry [31]. The resulting polymer exhibited an aminated-type (HN–C–NH–) framework instead of the expected imine (C=N–) configuration. This behavior aligns with imine–amine chemistry, where the imine double bond may react with primary amines to yield aminated functionalities [32]. Considerable advancements have been achieved in designing porous organic networks with diverse surface areas and functionalities through varied amine–aldehyde combinations within Schiff base frameworks [33–37]. Recently adapted Schwab's approach to synthesize crystalline imine-linked Schiff base polymers derived from tetraphenyladamantane [38]. These networks, tested at 77 K and 1 bar, exhibited BET surface areas up to 1.045 m²/g and hydrogen uptakes of 1.26 wt% [39].

2. Materials and Methods

2.1. Materials

Merck (Schnelldorf, Germany) provided the chemicals, reagents, and solvents, which were utilized exactly as they were delivered.

2.2. Physiochemical Measurements

The surface morphology of the synthesized Schiff bases was examined using field emission scanning electron microscopy (FESEM, TESCAN MIRA3, Kooutovice, Czech Republic) operated at an accelerating voltage of 10 kV. The N₂ adsorption–desorption isotherms were recorded at 77 K with a Quantachrome chemisorption analyzer (Quantachrome Instruments, Boynton Beach, FL, USA). Prior to measurement, samples A and D were degassed under vacuum for 6 hours at 100 °C while being purged with a continuous flow of nitrogen gas (Cascade TEK, Cornelius, OR, USA) to remove residual moisture and volatile species from the pores.

The specific surface area was determined using the Brunauer–Emmett–Teller (BET) equation at a relative pressure (P/P_0) of 0.98, and the pore size distribution was evaluated via the Barrett–Joyner–Halenda (BJH) method. Hydrogen adsorption measurements were

conducted at 50 bar and 323 K using a Hsorb 2600 high-pressure volumetric adsorption analyzer (Gold APP Instruments Corp., Beijing, China) equipped with dual degassing and analysis ports. Each hydrogen storage test was repeated at least ten times to ensure pressure optimization.

A known volume of hydrogen gas was introduced into the measurement cell containing the Schiff base sample until equilibrium between the adsorbed and free gas phases was achieved. The adsorbed gas quantity was calculated based on the recorded data and the final equilibrium pressure, automatically monitored by the instrument software.

2.3. Synthesis of Melamine- A–D Schiff Bases :

By condensing melamine with 3 molar equivalents of each of the following aromatic aldehydes: *para*-nitrobenzaldehyde, *ortho*-hydroxybenzaldehyde, *meta*-hydroxybenzaldehyde, and *para*-hydroxybenzaldehyde in boiling dimethylformamide under reflux for 6 hours., Schiff bases melamine-(A–D) were made using a previously described method Figure 1. Synthesis of Milamine-(A-D) Schiff base.[40]

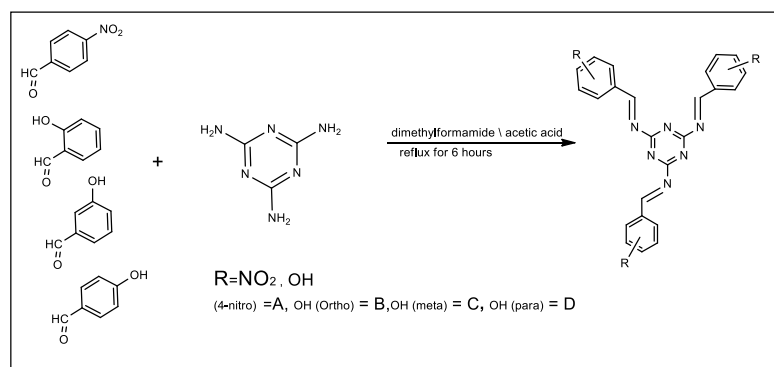


Figure 1. Synthesis of Schiff base Melamine-(A-D)

3. Results and Discussion

3.1. Synthesis of Schiff Bases Melamine-(A–D)

The physical characteristics of the synthesized Schiff bases Melamine - (A–D), such as melting points and hues, as well as the spectroscopic data from the ^1H -NMR and FT-IR spectra, were the same as those of the previously described bases [41,42]. The synthesized Schiff bases (melamine-A) chemical D's structures are shown in Figure 1. Nitro groups were present in Schiff bases (melamine –A), while variously arranged hydroxyl groups were present in Schiff bases (melamine- B-D). (*ortho*, *meta*, and *para*)

3.2. FESEM and EDX of Schiff Bases (melamine- A–D)

The surface morphologies of the synthesized Schiff bases (*melamine*-A–D) were examined using field emission scanning electron microscopy (FESEM) and energy-dispersive X-ray spectroscopy (EDX). The obtained micrographs revealed that the samples exhibited predominantly homogeneous and amorphous surfaces with micro-sized particles. The pore diameters of the Schiff bases ranged between 20 and 392 nm, with samples *melamine*-B and *melamine*-D showing the largest pore size as shown in Figure 2. Schiff base *melamine*-A displayed a distinct morphology compared to the others, which can be attributed to the presence of a nitro substituent. This substituent induced irregularity in particle size and shape, resulting in a highly porous architecture. The incorporation of nitrogen-rich functional groups is known to enhance surface area and hydrogen adsorption efficiency [43].

Schiff bases *melamine*-(A-D) had pores that were larger than those of telmisartan tin complexes and smaller than those that had been previously described for a number of POPs [44–46]. For example, POPs with 1,4-diaminobenzene-derived polyphosphates

displayed disordered and porous architectures with pore diameters of 50 –981 nm [47]. Additionally, porous structures with hole diameters ranging from 28 to 806 nm were visible in benzidine-derived polyphosphates [48].The pore size dimensions of telmisartan tin complexes, however, were measured within the interval of 20–51 nm.

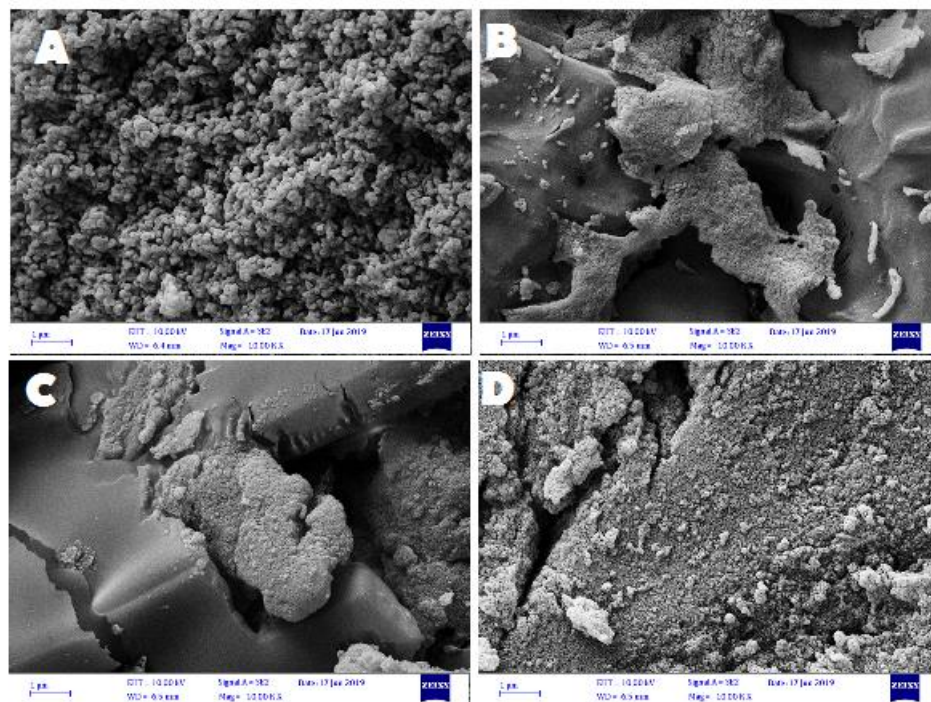


Figure 2. Field emission scanning electron microscopy (FESEM) image of Schiff base *melamine*-A,B,C,D

Figures shows the damages on the Schiff Bases *melamine*-(A–D) surfaces EDX spectroscopy was used as a reliable method to determine the elemental mapping of Schiff Bases *melamine*-(A–D). The EDX spectra of the Schiff Bases *melamine*-(A,B, C and D) showed high absorption bands, corresponding to the carbon, nitrogen and oxygen atom [49]. Figures . (3-6) as shown Schiff Bases *melamine*-(A,B,C , and D) in respectively suggestive of the presence of these elements.

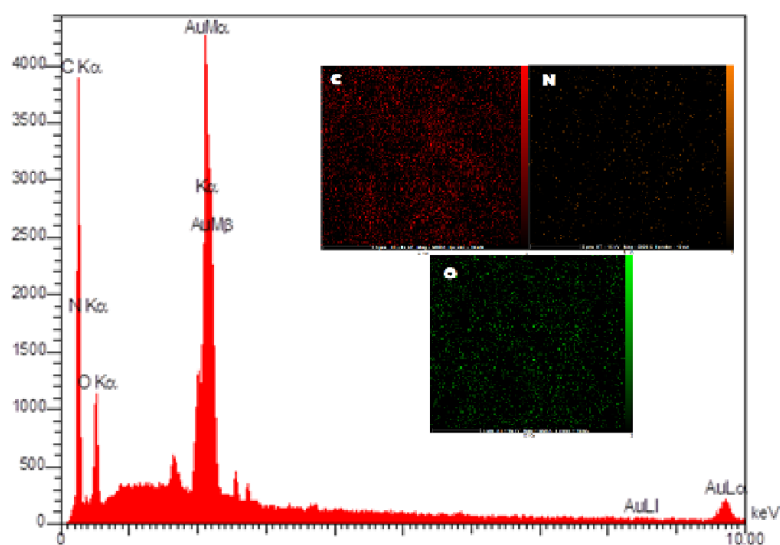


Figure 3. EDX spectra of Schiff base *melamine*-A

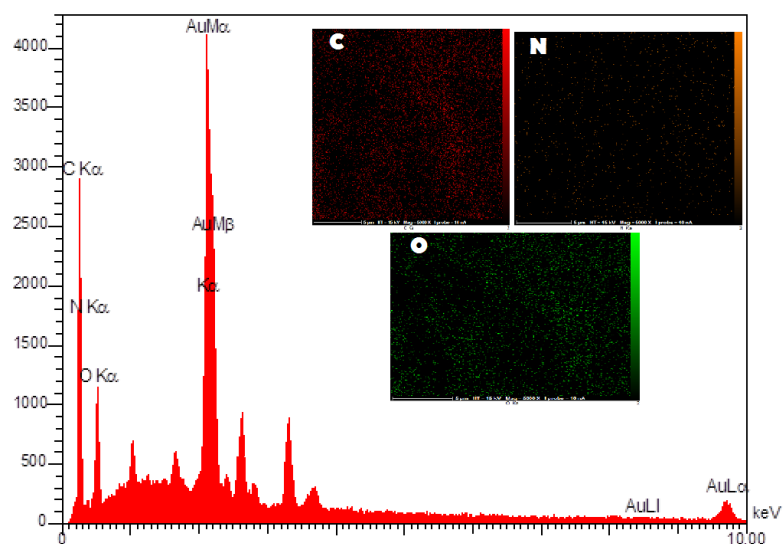


Figure 4. EDX spectra of Schiff base *melamine-B*

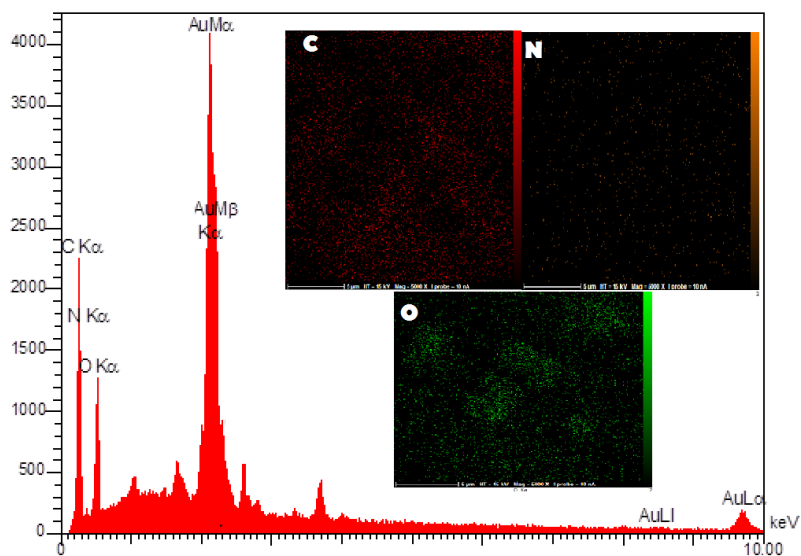


Figure 5. EDX spectra of Schiff base *melamine-C*

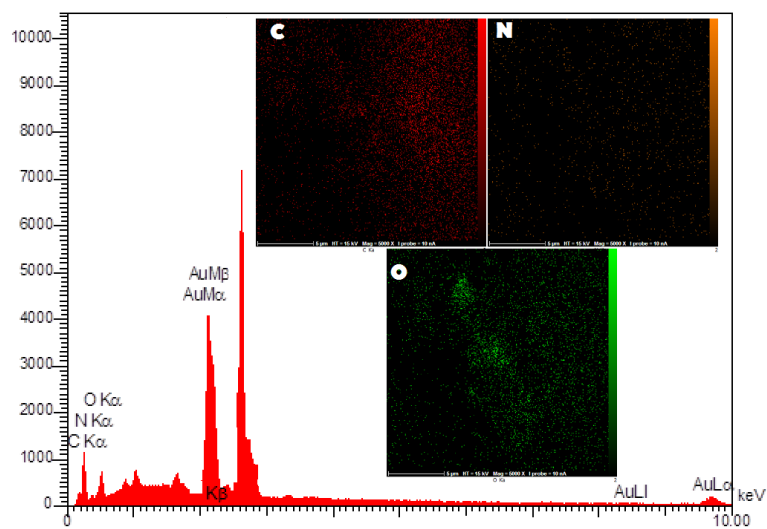


Figure 6. EDX spectra of Schiff base *melamine-D*

3.3. N₂ Adsorption–Desorption of Schiff Bases *melamine*-(A-D)

At 77 K, For the Schiff bases *melamine*-(A–D), measurements of N₂ adsorption and desorption were performed. The N₂ isotherm for A has a form similar to a type IV isotherm. The type III isotherm, in which monolayer formation was not seen, and the N₂ sorption isotherms in the Schiff bases *melamine*-(B–D) were essentially similar.[50]

The nitro group-containing Schiff base *melamine*-A, which was produced, had the highest pore volume (0.0361 cm³/gm) and surface area (SBET = 11.63 m²/g), but the smallest pore size diameter (1.70 nm). Compared to Schiff base *melamine*-(B–D) (which has a hydroxy group), Schiff base *melamine*-A was mesoporous. *ortho*, *meta*, and *para* orientations of the aryl ring) showed microporous structures (pore diameter: 2.440–3.631 nm). Some tin compounds and POPs showed similar-sized pores in their porous architectures.

In contrast to the 2.43 nm for telmisartan tin complexes, porous polyphosphates produced from either 1,4-diaminobenzene or benzidine, respectively, showed pore diameters of 1.96–2.43 nm [51] or 2.43–2.86 nm [52].

The amount of gas that was taken in was detected using a gravimetric approach, which allowed the gas adsorption isotherm to be calculated [53]. The gas's physisorption isotherms have also been calculated using the gas quantity that was removed from the gas phase. The pore size distribution can be determined by using the desorption or adsorption branch of the isotherm[54] as shown in Figures (7,8)

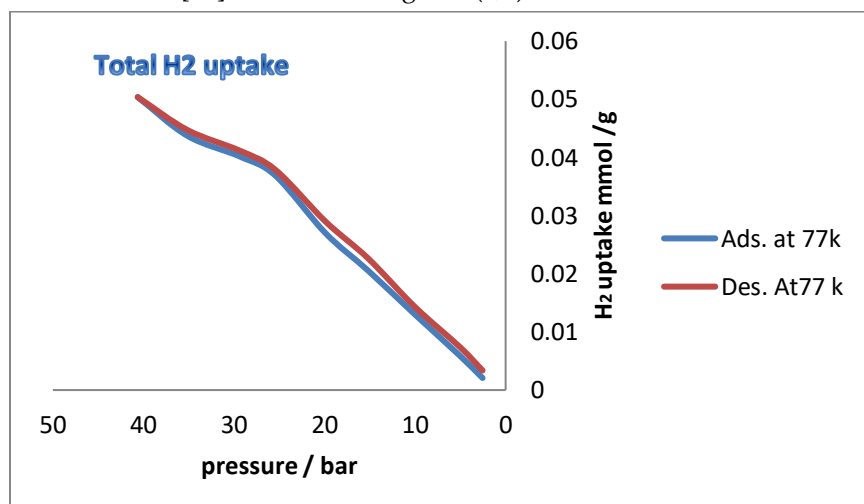


Figure 7. ADS, adsorption; DES, desorption Isotherm of H₂ for Schiff base *melamine*-A.

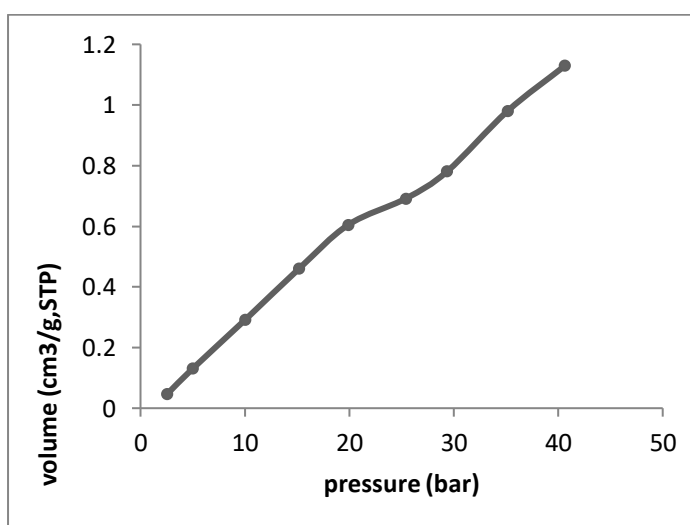


Figure 8. Volumetric of H₂ Uptake by Physical Adsorption for Schiff base *melamine*-A in (0-45 bar).

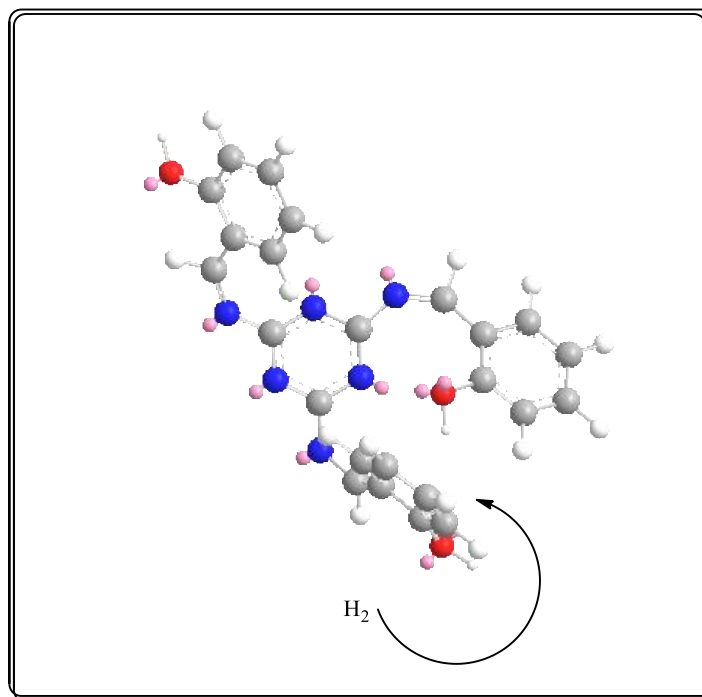


Figure 9. Structure for Schiff base *melamine-B* to Uptake of H_2

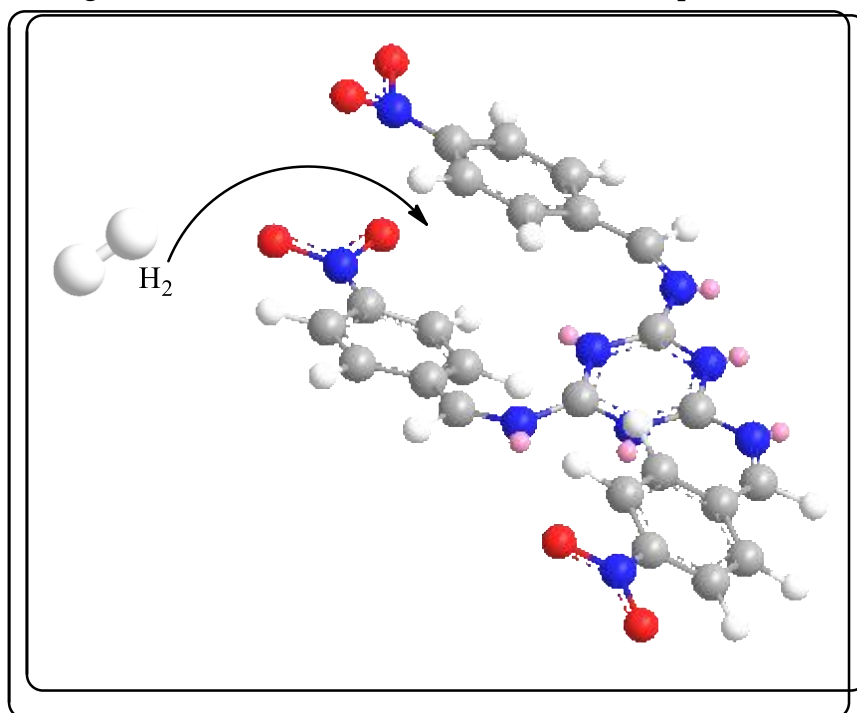


Figure 10. Structure for Schiff base *melamine-A* to Uptake of H_2

4. Conclusion

At 323 K as a possible H_2 storage media, four melamine Schiff bases have been investigated. 40 bars as well. With pore volumes ranging from (0.005-0.036 cm^3/g) and widths ranging from (1.67 to 3.353 nm), these compounds also have a comparatively low surface area (SBET = 4.9-10.8 m^2/g). The Schiff bases demonstrated significant H_2 uptake (0.002775 - 0.050377 mmol/g) and volume of H_2 uptake by Absorbed (0.062193 - 1.129143 $cm^3/g, STP$), presumably as a result of their high the presence of heteroatoms and aromaticity.

The Schiff base melamine with a *nitro* group showed the biggest effective H₂ absorption (0.050377 mmol/g) because of the high nitrogen (heteroatom) concentration in the porous material. Compared to Schiff bases containing nitro groups, those containing hydroxy groups substituent appear H₂ uptake (0.002775mmol/g). Its have bigger pore width but lower surface area and pore volume. Such Schiff base can be employed on an industrial scale because it is low cost and simple to produce in high yield.

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