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Effect of Semi-Closed Vessel on the Yield of g-C_3N_4 Prepared from Melamine and Melamine Cyanurate

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Optimization of the synthesis conditions for graphitic carbon nitride (g-C_3N_4) is a practical challenge aimed at maximizing product yield. The primary issues stem from the fact that the synthesis involves thermal polycondensation at temperatures of 500–600 °C, accompanied by precursor sublimation and oxidation, which significantly reduce the yield of the final product. In this study, the synthesis conditions of g-C_3N_4 were optimized using melamine and melamine-cyanurate under various atmospheric conditions and reactor configurations. It was established that synthesis in an open crucible in an oxygen-containing environment is the least effective, whereas conducting the process in a nitrogen atmosphere increases the yield. The best result was achieved using a semi-closed crucible without the need for a specialized atmosphere, which minimized the effects of sublimation and oxidation while offering a rational approach in terms of process efficiency.

Keywords: Graphitic carbon nitride, synthesis optimization, yield enhancement, thermal polycondensation, melamine, melamine-cyanurate.

Влияние полузакрытого тигля на выход g-C_3N_4, полученного из меламина и меламин-цианурата

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Оптимизация условий синтеза графитоподобного нитрида углерода (g-C_3N_4) представляет собой актуальную практическую задачу, направленную на достижение максимального выхода продукта. Основными проблемами является то, что синтез осуществляется посредством термической поликонденсации при температурах 500–600 °C, сопровождающийся процессами сублимации и окисление прекурсоров и приводящими к существенному снижению выхода конечного продукта. В настоящем исследовании были оптимизированы условия синтеза g-C_3N_4 с использованием меламина и меламин-цианурата при различных атмосферных условиях и конфигурациях реактора. Установлено, что синтез в открытом тигле в кислородсодержащей среде является наименее эффективным, тогда как проведение процесса в атмосфере азота увеличивает выход. Наилучший результат был достигнут при использовании полузакрытого тигля без использования специальной атмосферы, что обеспечило минимизацию эффектов сублимации и окисления, а также является рациональным с позиции эффективности проведения процесса.

Ключевые слова: Графитоподобный нитрид углерода, оптимизация синтеза, повышение выхода, термическая поликонденсация, меламин, меламин-цианурат.

Introduction

Graphitic carbon nitride (g-C_3N_4) is polymacrocyclic material containing heptazine heterocycles linked by aza- or imino-bridges (Figure 1). g-C_3N_4 is a metal-free, visible-light-active photocatalyst that has attracted significant attention for its potential in sustainable energy and environmental applications, including hydrogen evolution, pollutant degradation, CO_2 reduction.[1–4] Carbon nitride also can be used as a sensor for gases or substances like NO_2, CO or ractopamine.[5,6] Unique properties of g-C_3N_4, such as a moderate bandgap (~2.7 eV), high thermal and chemical stability, and tunable structure, make it an ideal candidate for photocatalysis.[7,8]

The synthesis of g-C_3N_4 typically involves thermal polycondensation of nitrogen-rich precursors, such as melamine, urea, or melamine-cyanurate (MCA), at temperatures of 500–600 °C.[8–11] During this process, precursors transform into heptazine-based intermediates, which polymerize to form the layered, graphitic structure of g-C_3N_4.[13] However, the synthesis is often plagued by low yields due to two primary challenges: sublimation of precursors at 300–350 °C and their oxidation in oxygen-containing media, leading to significant mass losses. The synthesis using melamine-cyanurate (MCA) is of particular interest due to the use of a pre-organized material structure, which results in a final product with high specific surface area and structural defects that positively influence its photocatalytic properties.[14] The issue of mass loss during the thermal polycondensation of MCA is more significant than with melamine, owing to the presence of cyanuric acid in the supramolecular complex, which actively decomposes into gaseous products.[15]

Despite extensive research on g-C_3N_4 synthesis, quantitative data on yields remain limited and inconsistent, with most studies focusing on improving photocatalytic performance rather than maximizing material yield. The highest reported yield of 61% was achieved under vacuum, but this method requires complex equipment, high energy consumption, and is not easily scalable for industrial applications.[16] An increase in product yield has also been observed when synthesis is conducted in a nitrogen or argon atmosphere.[17] However, this approach requires more expensive equipment and the use of gases, which limits the scalability of the process. A more attractive method, described by Liyan Wang, Yangwen Hou, and co-authors,[18] achieved a carbon nitride yield from melamine exceeding 50% by mass using a simple semi-closed crucible method. These limitations and opportunities underscore the importance of developing optimized synthesis protocols that can maximize product yield while preserving the structural and functional properties of g-C_3N_4, which is the primary focus of this study.

The objective of this study is to optimize the synthesis conditions of g-C_3N_4 to maximize yield using MCA as perspective precursor. Starting with a baseline synthesis in air using an open crucible, we systematically investigated the effects of nitrogen atmosphere and semi-closed crucible systems in a muffle furnace, drawing on literature insights. The semi-closed crucible approach aims to minimize sublimation by trapping precursor vapors while leveraging the uniform heating of a muffle furnace. By comparing different atmospheres and reactor configurations, this work seeks to develop a practical, scalable method that rivals vacuum-based yields without requiring specialized equipment. The findings contribute to bridging the gap between laboratory-scale synthesis and industrial applicability, offering a pathway to efficient g-C_3N_4 production for photocatalytic applications.

Experimental

The following reagents were used: melamine (99%, Macklin), cyanuric acid (98%, Macklin), dimethyl sulfoxide (DMSO, chemically pure, ECOS-1), and bidistilled water. Commercial g-C_3N_4 from Macklin was used as a reference material.

Synthesis of Melamine-Cyanuric Acid (MCA) Complex: MCA was synthesized by preparing 0.1 M solutions of melamine and cyanuric acid in dimethyl sulfoxide (DMSO). The solutions were mixed in equimolar amounts, heated to 80 °C, and stirred for 1 hour. The resulting precipitate was centrifuged, washed with water and ethanol, and dried in a vacuum oven at 80 °C for 2 hours. IR (KBr) ν cm^{-1}: 3391m, 3230m, 1781w, 1733s, 1662s, 1531s, 1445s, 1392w, 1086m, 1034w, 920m, 805w, 766s. According to SEM images the average particle size is about 3 μm.

Synthesis in Air Atmosphere: 1 g of melamine or melamine-cyanurate was placed in an open porcelain crucible and heated in a muffle furnace (EKPS-10) to 550 °C at a heating rate of 5 °C/min, with a holding time of 4 hours.

Synthesis in Nitrogen Atmosphere: 1 g of melamine or melamine-cyanurate was placed in a crucible in a tubular furnace. The reactor was purged with nitrogen (N_2), then heated to 550 °C at a rate of 5 °C/min under a continuous N_2 flow (10 mL/min) for 4 hours. The obtained material was washed 3 times with water and dried in a vacuum oven at 100°C under a pressure of 0.1 bar. The product yields under these conditions were 12.5% and 6.3% for g-C_3N_4 (melamine) and g-C_3N_4 (MCA), respectively.

Synthesis in Semi-Closed Crucible: 1 g of melamine or melamine-cyanurate was placed in a 25 mL crucible in a muffle furnace. Crucibles were sealed in three ways: with a lid, wrapped in aluminum foil (3 layers), or wrapped in foil with two 1 mm galvanized steel wire clamps. Heating: 550 °C at 5 °C/min, with a holding time of 4 hours. The obtained material was washed 3 times with water and dried in a vacuum oven at 100°C under a pressure of 0.1 bar. The product yields under these optimized conditions were 51% and 26% for g-C_3N_4 (melamine) and g-C_3N_4 (MCA), respectively. The specific surface area, measured by Brunauer-Emmett-Teller (BET) nitrogen adsorption-desorption analysis, was 18 m^2/g for g-C_3N_4 (melamine) and 142 m^2/g for g-C_3N_4 (MCA).

Microstructural analysis was performed using a Quattro S scanning electron microscope (SEM, Thermo Fisher Scientific). Samples were prepared by depositing a colloidal solution of particles in ethanol onto conductive indium tin oxide (ITO) glass, followed by complete drying. The specific surface area was measured using a Sorbi-MS instrument (ZAO META). Prior to analysis, samples were degassed at 100 °C under vacuum to remove adsorbed gases and moisture. Fourier-transform infrared (FTIR) spectra were recorded on an IRPrestige-21 spectrometer (Shimadzu) using KBr pellets, in the wavenumber range of 400–4000 cm^{-1} with a resolution of 2 cm^{-1}. X-Ray photoelectron spectroscopy (XPS) was conducted on a PREVAC EA15 spectrometer using Al Kα radiation (hν = 1486.6 eV, 150 W). Spectra were fitted using CasaXPS software.

Results and Discussion

Despite the extensive literature on the synthesis of g-C_3N_4, quantitative data on the yields of the material obtained through thermal polycondensation remain limited and inconsistent. Therefore, the primary goal of this study

was to identify optimal synthesis parameters to maximize the yield of graphitic carbon nitride.

Three synthesis approaches were investigated: (1) open crucible in air, (2) nitrogen atmosphere in a tubular furnace, and (3) semi-closed crucible in a muffle furnace (Figure 1). Melamine and melamine-cyanurate (MCA) were used as precursors, with synthesis conducted at 550 °C, a heating rate of 5 °C/min, and a 4-hour holding time.

As a starting point, the simplest synthesis method was selected: heating melamine to 550 °C in an air atmosphere with a holding time of 4 hours and a heating rate of 5 °C/min. The synthesis was conducted in an open porcelain crucible. Experiments showed that, with a 1 g melamine loading, the yield of the product was only 5 mg, corresponding to 0.5% of the initial mass. In contrast, the use of melamine-cyanurate (MCA) as a precursor with the same 1 g loading did not yield any significant amount of product under these conditions (Table 1).

In a second approach, g-C_3N_4 synthesis was conducted *via* thermal treatment in an inert nitrogen (N_2) atmosphere. The precursors, melamine and MCA, were placed in crucibles within a tubular furnace. The reactor was purged with nitrogen, followed by heating to 550 °C at a rate of 5 °C/min under a continuous N_2 flow of 10 mL/min for 4 hours. The yield of g-C_3N_4 was 12.5% by mass for melamine and 6.3% for MCA, relative to the initial precursor mass (Table 1).

Table 1. Yields of g-C_3N_4 under different synthesis conditions.

Synthesis conditions	Yield, %	
	Melamine	MCA
Open crucible, air	0.5	0
Nitrogen atmosphere, tubular furnace	12.5	6.3
Semi-closed crucible (lid)	36	17
Semi-closed crucible (foil)	45	23
Semi-closed crucible (foil + wire)	51	26

This represents a clear improvement in yield compared to synthesis in air, attributed to the prevention of oxidative degradation. However, the yields remained significantly lower than the record 61% reported by Yuan for vacuum-based synthesis.[16] The primary limitation in this setup was identified as the sublimation of the precursors which occurs at temperatures between 300–350 °C. Consequently, significant mass losses were observed due to precursors escaping the crucible. Visual inspection of the reactor revealed substantial deposition of precursors on the walls of the glass tube (Figure S1), confirming sublimation losses. A critical drawback of the tubular furnace design is the uneven heating, with cooler tube ends promoting precursor condensation and crystallization on the reactor walls. Based on the IR and XRD spectra of the samples obtained in a nitrogen atmosphere (Figures S3-4) the intended synthesis temperature regime was not maintained due to the cooling of the reaction mass by the incoming nitrogen. The resulting materials correspond to g-C_3N_4 synthesized at temperatures of 450–500 °C, and the structure was not fully formed.[19]

While the use of an inert atmosphere increased the yield compared to air, the persistent issue of sublimation prevented satisfactory results. Consequently, further exploration of synthesis methods was pursued, with attention focused on the semi-closed crucible approach described in prior work.[20] Experiments conducted in the tubular furnace revealed significant material loss due to sublimation during the reaction. Additionally, the initial configuration of the tubular furnace exhibited clear limitations: a thermocouple positioned inside the tube near the crucible is necessary, and the inert gas should be preheated to reduce sublimation. Based on these findings, the decision was made to revert to using a muffle furnace as the heating device. The muffle furnace offers an advantage over the tubular furnace in maintaining uniform temperature distribution. However, it has a drawback: the available furnace configuration lacked the capability to introduce inert gas into the heating zone. Synthesis in air, as noted in the literature,[21] significantly reduces the yield of the target product due to the oxidation of precursors by atmospheric oxygen, forming complete and incomplete combustion

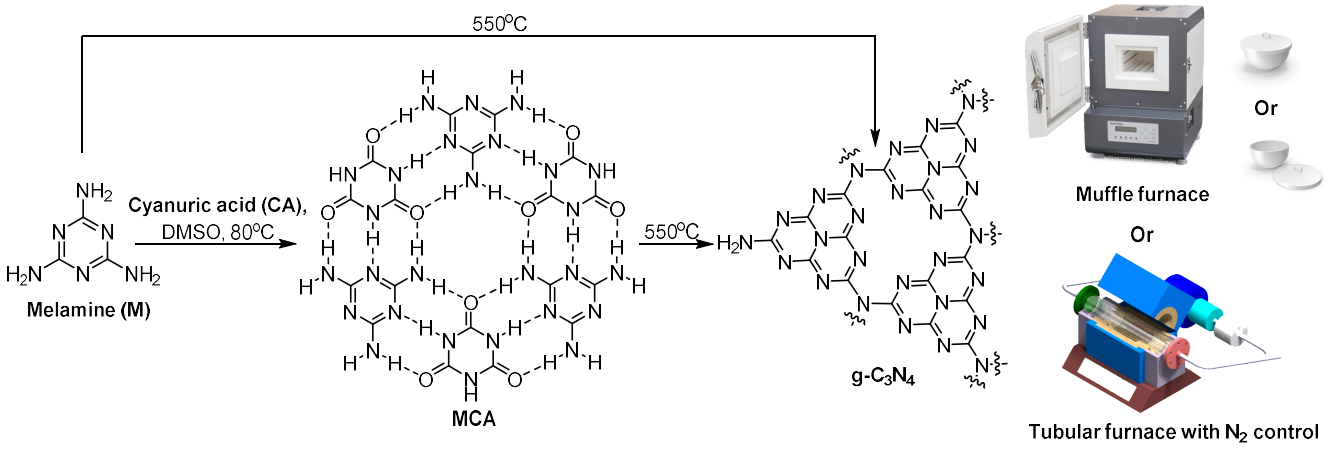

Figure 1. Methods of synthesis of g-C_3N_4.

products. To address this, a series of syntheses were conducted with various crucible sealing methods for the preparation of g-C_3N_4 from melamine based on data from the semi-closed crucible synthesis study.[20]

The semi-closed crucible method in a muffle furnace, using various sealing techniques (lid, foil, foil with wire clamps), significantly improved yields. The highest yield of 51% was achieved for melamine with foil and wire sealing, which minimizes sublimation by trapping precursor vapors and creating a local inert atmosphere via evolved gases (NH_3, CO_2) (Table 1) For MCA the optimal yield was 26% with the same sealing, limited by CO_2-induced structural disruptions.[15] This indicates that the use of foil and wire minimizes precursor losses and enhances the yield of the final product. The crucibles were sealed with three layers of aluminum foil and two clamps made of 1 mm galvanized steel wire (Figure S2).

Infrared (IR) spectroscopy was employed to investigate the chemical structure and bonding characteristics of the synthesized g-C_3N_4 materials. The IR spectrum of the synthesized g-C_3N_4 was compared to commercial g-C_3N_4 from Macklin, used as a reference material. For g-C_3N_4 derived from melamine under the optimized semi-closed crucible conditions (foil with wire clamps), the IR spectrum (Figure 2) exhibits a close match with that of commercial graphitic carbon nitride. Characteristic absorption bands include a sharp peak at 810 cm^{-1}, corresponding to the breathing mode of the tris-triazine ring, a broad region between 1200 and 1600 cm^{-1} attributed to the stretching vibrations of C-N and C=N bonds, and a band at 3000–3500 cm^{-1} associated with N-H stretching from uncondensed amino groups. g-C_3N_4 synthesized from melamine-cyanurate (MCA) under the same conditions shows distinct variations in its IR spectrum. The 1200–1600 cm^{-1} region exhibits enhanced intensity, reflecting increased structural defects and porosity resulting from CO_2 release during cyanuric acid decomposition. The 3000–4000 cm^{-1} region shows broader N-H and O-H stretching of surface groups due to higher specific surface area.

X-Ray diffraction (XRD) analysis was conducted to evaluate the crystallographic structure of the synthesized g-C_3N_4 materials, providing insights into their crystallinity and interlayer ordering. The XRD patterns of g-C_3N_4 derived from melamine under the optimized semi-closed crucible conditions (foil with wire clamps) fully correspond to those of commercial graphitic carbon nitride (Figure 3). A sharp and intense (002) peak is observed at 27.4°, corresponding to an interlayer d-spacing of approximately 0.326 nm, which reflects the characteristic stacking of two-dimensional tris-triazine layers, consistent with highly crystalline g-C_3N_4. This alignment with commercial standards confirms the effectiveness of the semi-closed crucible method in producing a structurally robust material. In contrast, g-C_3N_4 synthesized from melamine-cyanurate (MCA) under the same conditions exhibits notable differences in its XRD pattern, attributed to its more porous and disordered structure. The (002) peak is broader and less intense, appearing at a slightly shifted position (27.23°), with a reduced d-spacing resolution, indicating lower crystallinity and increased structural defects. This is consistent with the release of CO_2 during cyanuric acid decomposition, which disrupts the ordered stacking of graphitic layers and increases porosity. The (100) peak, associated with in-plane packing, is consistently labeled at 12.75° across all three samples, which may indicate a uniform lateral ordering despite the differences in interlayer structure.

X-ray photoelectron spectroscopy (XPS) was used to analyze the surface chemical composition and bonding states of the g-C_3N_4 material, with a focus on the sample derived from melamine-cyanurate (MCA) (Figure 4).

The overview spectrum (Figure 4a) reveals the elemental composition of the MCA-derived g-C_3N_4, with contents of 40.68% carbon (C), 57.12% nitrogen (N), and 2.21% oxygen (O), indicating a predominantly carbon-nitrogen framework with minor oxygen incorporation. In the C 1s spectrum (Figure 4b), the primary peak at 288.0 eV is assigned to N-C=N and N-$(C)_3$ bonds, characteristic of the heptazine-based structure of g-C_3N_4. A secondary peak at 286.5 eV, with an intensity of 12.6%, is attributed to C-O and C-NH bonds, suggesting the presence of oxygen-containing defects or surface groups. The peak at 284.7 eV, typical of sp^2-hybridized carbon in C-C or C=C bonds, is indicative of structural defects within the g-C_3N_4 framework. Additionally, a peak at 289.3 eV corresponds to C=O bonds, likely from carboxylic groups (O-C=O), which may arise from surface oxidation.

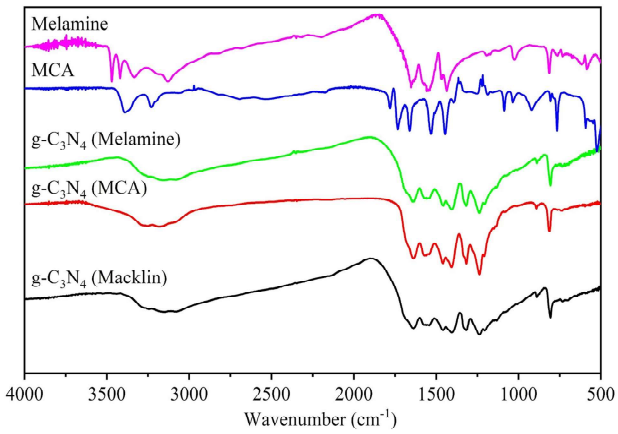

Figure 2. FTIR spectra of obtained g-C_3N_4.

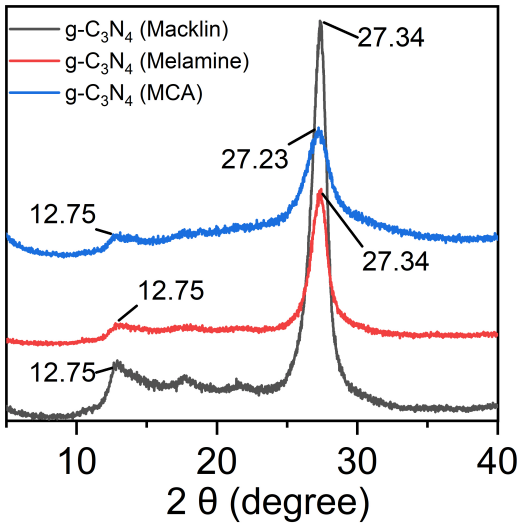

Figure 3. XRD of obtained g-C_3N_4.

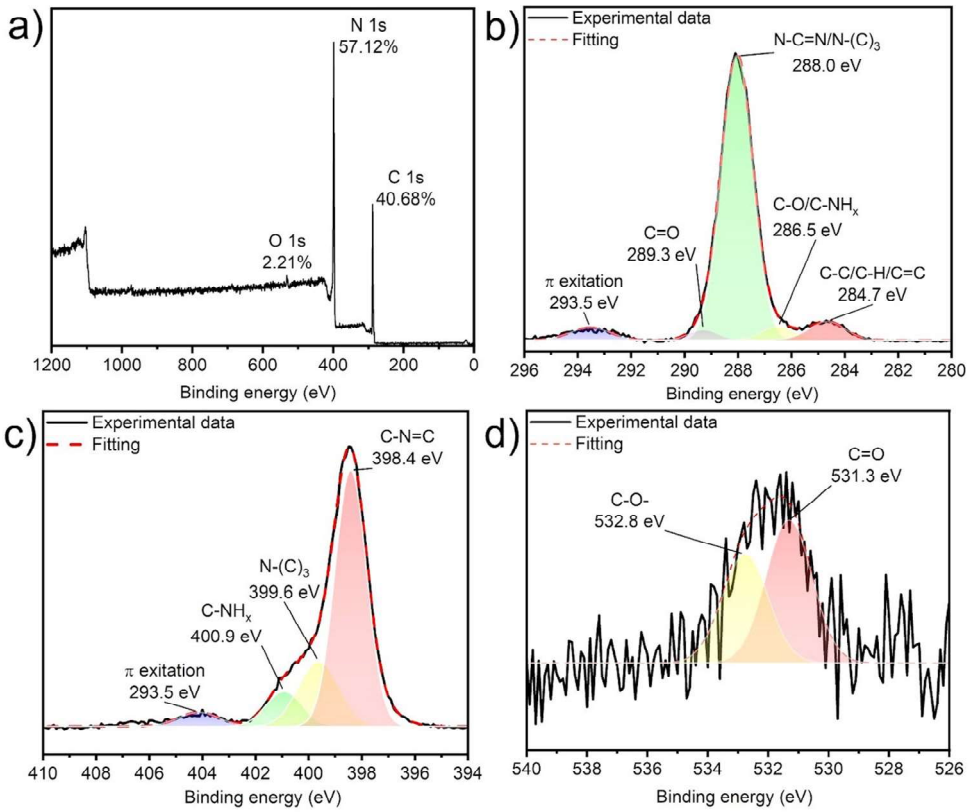

Figure 4. XPS of g-C_3N_4 (MCA): a) survey spectra, b) C 1s high-resolution spectra, c) N 1s high-resolution spectra, d) O 1s high-resolution spectra.

The N 1s spectrum (Figure 4c) shows a dominant peak at 398.4 eV, corresponding to C=N-C bonds within the heptazine units of g-C_3N_4. A peak at 399.6 eV is associated with N-$(C)_3$ bonds, while a peak at 400.9 eV indicates C-NH bonds linked to terminal amino groups on the material's surface. The O 1s spectrum (Figure 4d) features a peak at 532.8 eV, attributed to oxygen in C-O bonds, and a component at 531.3 eV, assigned to oxygen in N-C=O bonds, suggesting the presence of carbonyl functionalities or oxidized areas within the g-C_3N_4 structure.

Scanning electron microscopy (SEM) images revealed distinct morphological differences between g-C_3N_4 synthesized from melamine and melamine-cyanurate (MCA). The melamine-derived g-C_3N_4 consists of agglomerated particles with a broad size distribution ranging from 1 to 40 μm, lacking a defined morphology, which suggests uncontrolled aggregation during thermal polycondensation (Figure 5a). In contrast, g-C_3N_4 derived from MCA exhibits a uniform spherical morphology with an average particle size of approximately 3 μm, as determined by analyzing multiple SEM fields (Figure 5c). Notably, the MCA-derived g-C_3N_4 retains the spherical morphology of the MCA precursor (Figure 5b), indicating that the hydrogen-bonded self-assembly in MCA templates the structure of the final material. This preservation of morphology is consistent with literature reports, where hydrogen-bonded precursors like MCA direct the formation of ordered nanostructures in g-C_3N_4.[22]

The specific surface area of the synthesized g-C_3N_4 materials was determined using nitrogen adsorption-desorption analysis. According to the BET data, the specific surface area was 18 m²/g for g-C_3N_4 derived from melamine, 142 m²/g for g-C_3N_4 derived from MCA, and 16 m²/g for the commercial Macklin g-C_3N_4. These values highlight the significantly higher porosity and surface area of the MCA-derived material, which can be attributed to the structural disruptions caused by CO_2 release during synthesis, enhancing its potential for applications requiring high surface reactivity, such as photocatalysis. In contrast, the melamine-derived and commercial samples exhibit lower surface areas, consistent with their more ordered, less defective structures.

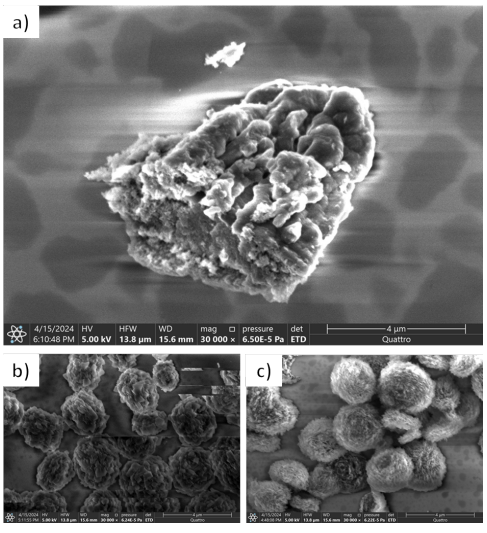

Figure 5. SEM images: a) g-C_3N_4 (melamine), b) MCA, c) g-C_3N_4 (MCA).

Conclusions

Various approaches to the synthesis of g-C_3N_4 were thoroughly explored in this study, revealing critical insights into the synthesis process. It was demonstrated that the primary causes of material loss and reduced yield stem from the oxidation of precursors and their sublimation, which significantly diminish the efficiency of the reaction. Although synthesis in an inert atmosphere effectively shields precursors from oxidative degradation, the persistent issue of sublimation continues to pose a substantial challenge. The adoption of a semi-closed crucible method marks a pivotal advancement, effectively counteracting the sublimation effect through the creation of a confined atmosphere that retains volatile precursors, while simultaneously preventing their exposure to oxidative conditions. During the synthesis of g-C_3N_4, the substantial release of gases such as NH_3 and CO_2 plays a crucial protective role, forming a localized inert environment that protects the material against oxidation throughout the process.

A particularly noteworthy finding is the significant influence exerted by the method of crucible sealing on the overall yield. The degree of sealing proves to be a decisive factor, with more robust and secure closures consistently correlating with higher yields of the final product, underscoring the importance of minimizing vapor loss. Under the most favorable conditions identified, the yield of g-C_3N_4 derived from melamine achieved 51% and for MCA 26% by mass, reflecting the efficacy of the optimized approach.

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