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SYNTHESIS AND STUDY OF STRUCTURAL, THERMAL AND COLLOIDAL PROPERTIES OF O-g-C_3N_4 NANOPARTICLES

Gozzal Sidrasuliyeva[1], Nuritdin T. Kattaev[1], Khamdam Akbarov[1], Rhayxona Muyassarova[1], Sarvar Parmonov[2], Sarvar Qorayev[3], Sadatdin Tileubayev[4], Erkin Ruziyev[5], Jamshid Ruziyev[5], Nodirbek Usmonov[6], Murodjon Samadiy[7], and Dilbar Ramazonova[7,✉]

[1]National University of Uzbekistan named after Mirzo Ulugbek, Uzbekistan
[2]Almalyk State Technical Institute, 110100, (Almalyk) Uzbekistan
[3]Denov Institute of Entrepreneurship and Pedagogy, Surkhandarya region, Denau, Street Sh. Rashidov, 360, Denau, 190506, Uzbekistan
[4]Karakalpak State University named after Berdakh, 230113, Uzbekistan
[5]Department of Analytical Chemistry, Samarkand State University named after Sharof Rashidov, 15 University Blvd, 140104, (Samarkand), Uzbekistan
[6]Fergana State Technical University, Fergana, 150109, Uzbekistan
[7]Department of Chemical Engineering and Biotechnology, Karshi State Technical University, Karshi, 180100, (Karshi), Uzbekistan
✉Corresponding author: dinaramazanova126@gmail.com

ABSTRACT

This research focuses on (O-g-C_3N_4), O-doped graphitic carbon nitride produced using melamine through a thermal polycondensation method under atmospheric conditions, followed by characterisation for structural, thermodynamic, and colloidal studies. The structure determination was accomplished through the FTIR and XRD analyses. XRD spectra reveal characteristic peaks of g-C_3N_4 with diffraction angles of about $2\theta \approx 13{,}27°$ and 27,17°, while crystallite size lies within 4-6 nm. DLS analysis reveals the hydrodynamic diameter on the order of 30 nm. Thermogravimetric analysis revealed that the decomposition process took place during the main stage within the temperature range of 542-684 °C, with a mass loss of ca (82 %). Graphite-type carbon nitride (g-C_3N_4) represents an interesting photocatalytic system in terms of non-metallic catalysts. The O-g-C_3N_4 was synthesised through polycondensation of the melamine at 500 °C temperature. The structural analysis was done using FTIR and XRD analyses. DLS and ζ-potential were used for colloidal analysis. X-ray phase analysis revealed characteristic reflections at $2\theta \approx 13.27°$ and 27.17°, with the crystallite size being 4-6 nm, the average particle size being approximately 30 nm, and the ζ-potential value of −17 mV indicating moderate colloidal stability of the system.

Keywords: Melamine; O-g-C_3N_4, Polycondensation, Nanoparticles, Thermal Analysis, Z-Potential, Colloidal Stability

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INTRODUCTION

The intensive development of industry goes hand in hand with the emergence of environmental issues related to pollution and energy resource depletion. In this connection, there is significant interest in methods for cleaning the environment and using renewable energy sources.[1-4] Photocatalysis may be considered one of the possible ways to address these environmental issues by harnessing solar energy to degrade organic substances into environmentally friendly materials.[5-8] Over the past few years, a number of semiconductor photocatalysts to catalyse chemical reactions have been discovered. One such material that merits mentioning is g-C_3N_4; it is an organic-inorganic free semiconductor photocatalyst possessing a graphite-layered structure with a band gap energy of 2.7 ev).[9-14] Moreover, this material is known to be chemically and thermally stable and features available starting materials.[15-19] The objectives of this research work are the synthesis of O-g-C_3N_4 using melamine and examination of the structure and colloidal properties of the synthesised material.

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EXPERIMENTAL

The initial substance was melamine ($C_3H_6N_6$). Graphite-like carbon nitride doped with oxygen (O-g-C_3N_4) was polycondensed at the temperature of 500 °C using melamine in the air for 3 hours.[20,21,22,23] The obtained substance was subjected to ultrasonic dispersion in a water–alcohol mixture using an ultrasonic disperser (50 kHz) to prepare a nanosuspension (Fig.-1). The FTIR spectra and X-ray analysis were done on the sample powders with CuKα radiation (λ = 0.15418 nm) to test structural properties. The sizes of the crystallites were determined based on the Scherrer formula.[24,25] The thermal stability using the thermogravimetric analysis was done and the scanning calorimetry on a Setaram LabSys Evo DTA/DSC +1600 derivative analyzer within the temperature between 30-700 °C at 10 °C/min heating rate. The size of particles in the dispersion medium was determined using dynamic light scattering, and its colloidal stability was evaluated in terms of the ζ-potential.

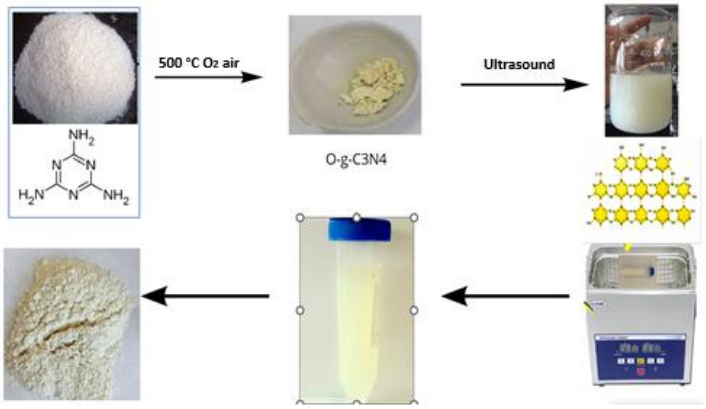


Fig.-1: Receipt Diagram O-g-C_3N_4

However, it is necessary to highlight that the selected method of thermal polycondensation of melamine under air conditions has a number of significant benefits compared to other ways of synthesising carbon nitride. It can be processed in a single step and only with a readily available starting material, without any other substances. Moreover, oxygen incorporation into the reaction product results in the generation of oxygen functional groups and defects in the g-C_3N_4 structure, which might cause some drastic changes in the physical characteristics of the material and also chemical characteristics. The chosen temperature range allows forming the heptazine structure and creating nanocrystals, and ultrasonic dispersion allows the formation of nanoparticles. The proposed approach, therefore, ensures that it is possible to fabricate oxygen-containing graphite-like carbon nitride with nanostructured characteristics and with colloidal stability under relatively simple conditions.

RESULTS AND DISCUSSION

Fourier transform infrared spectroscopy (Figure-2) was used to identify the structure of the formed compound. As one can see from the figure, there is a broad band at the position of 3157 cm^{-1}, meaning the presence of vibration of O-H and N-H bonds.

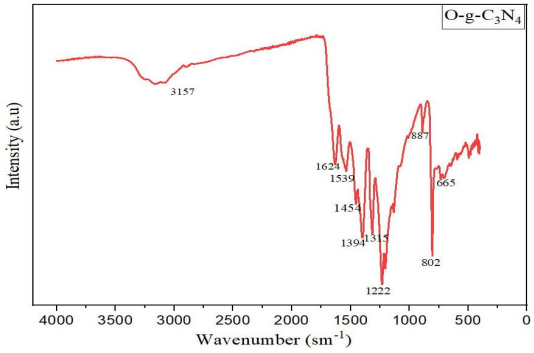


Fig.-2: IR Spectrum O-g-C_3N_4

Bands in the range of 1200- 1600 cm^{-1} correspond to vibrations of C=N and C-N bonds in the heptazine framework, which proves successful polymerisation. The presence of strong absorption peaks in this area is typical for the condensed g-C_3N_4 structure. The peak detected near 802 cm^{-1} is associated with the vibrations of the heptazine/triazine unit, which indicates the formation of a graphite-like structure. The results indicate that the oxygen-doped graphite-like carbon nitride was synthesized.

The crystal phase and structure of the material were studied with the X-ray diffraction. As depicted in Figure- 3, it is evident that there are reflections corresponding to the plane angles of 2θ = 13.27° and 27.17°, corresponding to the (100) and (002) planes of g-C_3N_4, respectively.

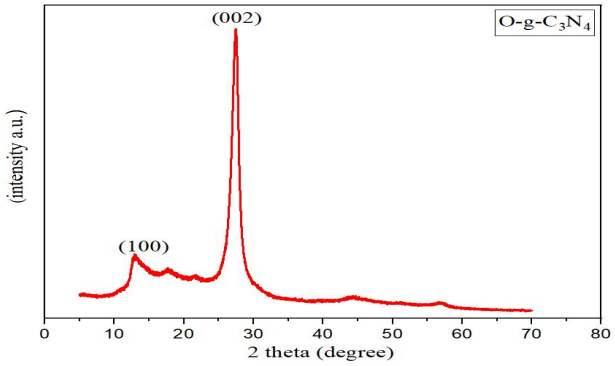


Fig.-3: Diffraction Pattern for O-g-C_3N_4

A diffraction peak around 2θ = 13.27° corresponds to the packing of heptazine structural units in the layer plane, which gives information on the structural ordering of the substance in the layer plane. The diffraction peak around 2θ = 27.17° is related to the interlayer spacing of the stacked heptazine layers.

Table-1: Crystallite Size O-g-C_3N_4

2θ(°)	d (Å)*	I/I_1	B (FWHM)	D, nm
13.2734	6.66503	34	1.384	6.04
24.9711	3.56301	18	1.455	5.84
27.1746	3.27890	100	2.181	3.92
29.5239	3.02311	13	1.500	5.72
77.4268	1.23164	10	0.572	18.59

Determination of the crystallite sizes based on the application of the Scherrer formula revealed the presence of the main peak at 4-6 nm, indicating the creation of the nano-sized crystal structure of the doped carbon nitride (Table-1). The thermal stability of the obtained compounds was estimated with thermal analysis, which is commonly applied for studying the stability of various inorganic and organic materials, such as carbon nitride with graphite-like structure.[26] The derivatogram for the O-g-C_3N_4 sample is presented in Fig.-4 and encompasses the temperature graph (T), thermogravimetry graph (TG), differential thermogravimetry graph (dTG), and differential scanning calorimetry (DSC) graph. The analysis of thermograms indicates that within the temperature interval of 56-531 °C, there is an endothermic phenomenon with a small reduction in mass according to the DSC graph. In this region, the thermal effect magnitude is 1.46–3.22 μV•s/mg, which is apparently associated with the removal of adsorbed moisture, dehydration, and partial structural rearrangement of the material. The most active process of thermal decomposition takes place at temperatures between 542-684 °C, when a considerable mass loss of the substance under investigation occurs. As a result of thermogravimetric analysis, it has been found that the total mass loss in this range equals 82%. Moreover, in the temperature range of 600-700 °C, mass loss increases up to 1.50-2.16 mg/min, indicating the destruction of a polymeric substance. Finally, as far as DSC measurements go, it has been established that the thermal effect amounts to 1.21-1.65 μV•s/mg. The oxygen introduced into g-C_3N_4 results in modification of its electronic structure, accompanied by the creation of defects in g-C_3N_4, which may cause alteration of the thermal behaviour of the polymer matrix.

Curve 1-temperature (T); Curve 2-thermogravimetric analysis (TGA/TG); Curve 3-differential thermogravimetry (DTG/dTG); Curve 4-differential scanning calorimetry (DSC/DSC). The experimental results match previous research data, by shows thermal decomposition of g-C_3N_4 takes place at 500-700 °C due to the destruction of condensed heptazine rings and C-N bond rupture in the polymeric matrix.[27]

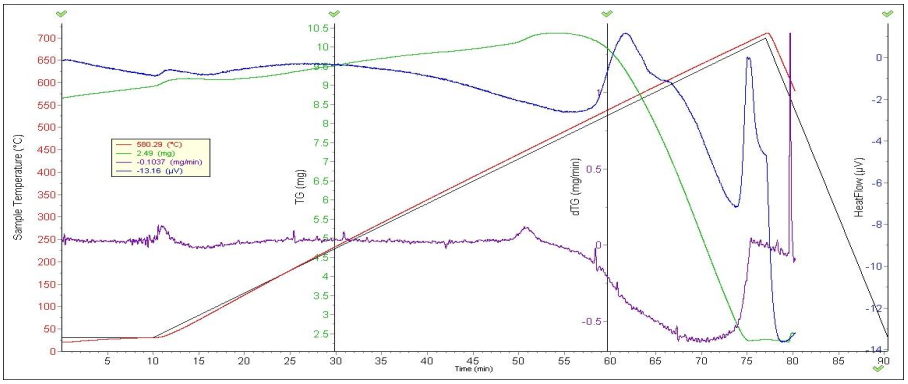


Fig.-4: Derivatogram of the Sample O-g-C_3N_4

Therefore, the outcome of the thermogravimetric analysis demonstrates high thermal stability of the obtained oxygen-doped carbon nitride-like graphene, and the main period of its decomposition at temperatures from 542 to 684 °C corresponds to the decomposition of the heptazine structure of the material. The size characteristics of the synthesized material particles were studied using dynamic light scattering.[28] The results show that the majority of the particles have an average hydrodynamic size of approximately 30 nm, indicating the formation of a nanodispersed system (Fig.-5).

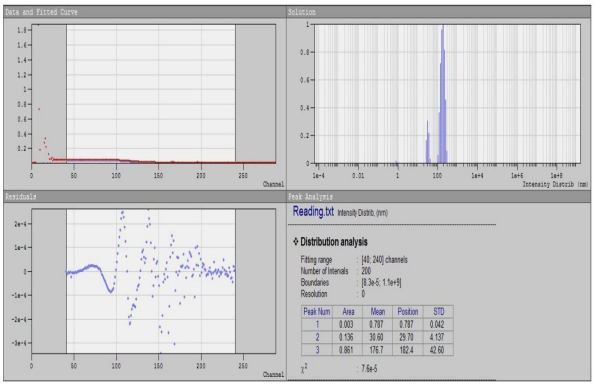


Fig.-5: Characteristic dimensions O-g-C_3N_4

As should be pointed out, the value of the hydrodynamic radius of particles calculated using this method can be larger than the values for the size of crystallites obtained from the analysis of X-ray diffraction data (4-6 nm). The reason for this lies in the fact that, during dynamic light scattering, not only the size of the particle is estimated but also the size of its solvation shell.

The following was used to evaluate the stability of the resulting colloid system: the value of the ζ-potential (Fig.-6). The resulting value of the ζ-potential for O-g-C_3N_4 equals -17 mV. The existence of the negative surface charge is described by the presence of the following groups: –NH, –NH_2, C=N, and the release of protons from the surface centres. The formation of a negatively charged surface leads to electrostatic repulsion between particles, which prevents their intense aggregation and helps maintain the dispersed state of the system.

CONCLUSION

In the current work, O-g-C_3N_4 was produced under ambient conditions by a thermally assisted polycondensation reaction of the melamine molecule. The structural property investigations have established that the material produced had the lamellar structure of graphitic carbon nitride, where crystallites were in the range of 4 to 6 nm, and 30 nm mean hydrodynamic particle.

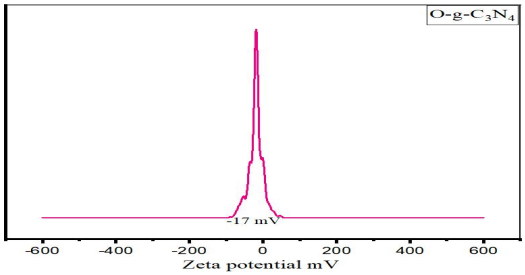


Fig.-6: ζ-Potential O-g-C_3N_4

The thermal studies have shown that the large proportion of degradation occurred at temperatures between 542-684 °C with the total mass loss of approximately 82%. The particle surface was found to be negatively charged (ζ-potential of about -17 mV), which guaranteed moderate colloidal stability of the dispersion. The synthesis technique suggested is easy and technologically viable as it allows the generation of O-g-C_3N_4 utilising a readily available and commercially cheap material, melamine, in one step without the utilisation of extra reagents. The nanoscale form, surface functional groups, and stable dispersion of the particles provide good conditions for the eventual utilisation of the resulting material in the photocatalytic systems.

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CONFLICT OF INTERESTS

The authors state that there are no conflicts of interest in this research.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-9: Industry, Innovation and Infrastructure*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

Gozzal Sidrasuliyeva https://orchid.org/0009-0002-0887-272X
Nuritdin T. Kattayev https://orchid.org/0000-0002-0276-2717
Khamdam Akbarov https://orchid.org/0000-0002-3225-2427
Rhayxona Muyassarova https://orchid.org/009-0008-2994-3631
Sarvar Parmonov https://orchid.org/0000-0001-6634-8606
Sarvar Qorayev https://orchid.org/0009-0004-3219-5102
Sadatdin Tileubayev https://orchid.org/0009-0006-0047-737X
Erkin Ruziyev http://orchid.org/0000-0002-1043-0528
Jamshid Ruziyev http://orchid.org/0000-0001-7623-3343
Nodirbek Usmonov https://orchid.org/0009-0003-8648-4025
Murodjon Samadiy https://orcid.org/0000-0003-4467-291X
Dilbar Ramazonova https://orcid.org/0000-0002-0232-0185

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