N-graphene synthesized in astrochemical ices

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Abstract. In our quest for the presence of large complex molecules containing a majority of carbon in the interstellar medium (ISM), the search for graphene plays a central role due to its nature in making other carbon structures. Although the ingredients for graphene synthesis are present in the ISM, conclusive laboratory evidence of such formation is lacking. Therefore, in our laboratory experiments simulating the cold ISM conditions, we subjected icy mantles of benzonitrile, an aromatic with a cyanide side chain that has recently been detected in the interstellar medium, to vacuum ultraviolet photon irradiation. The irradiated ice was observed to leave a residue upon warming to room temperature. The residue was removed from the substrate and placed on a Quantifoil grid for electron microscopy analysis. Transmission electron microscopy showed quantum dots (QD) and nitrogen-doped graphene (N-Graphene) sheets. Diffraction and energy-dispersive X-ray spectroscopy revealed the crystalline nature and carbon–nitrogen composition, of the observed graphene sheet. This is the first evidence of QD and N-graphene synthesis in ice irradiation at interstellar temperatures.

1 Introduction

Cold ice covered dust particles in the interstellar medium (ISM) are known to be the tiny chemical factories responsible for the evolution of complex molecules observed in the ISM [1–3]. The molecular complexity of the icy mantles on these cold dust grains is driven by energetic particles irradiation and temperature changes. Although the molecular ices present as icy mantles on a dust grain are volatile, the change in physico-chemical nature brought by the irradiation can initiate the growth of larger molecules, leading to nucleation [4], producing refractory residues that may be observed at higher temperatures when simpler species are sublimed from the surface. Spectral signatures of such a residue obtained from a mixture of organic and inorganic molecular ice [5] have been used to explain the 3.4 µm signature in ISM clouds from space-based observations.

The nature of the residue depends on the chemical composition of the icy mantle undergoing irradiation under conditions commensurate to those found in the ISM and in the solar system [6]. Most of the residues are complex organic [4, 7–12] and inorganic [13–17] compounds. For cometary ices present in the Oort cloud, such residues may form an outer web of

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a non-volatile coating [18, 19] preventing further ice irradiation. Indeed the recent discovery of an amno-
minated refractory residue [20, 21] on comets may be a result of ice irradiation. An icy mixture containing sim-
ple ices, upon energetic processing, may also leave bio-
residues [22] that might be of interest in the Origin of Life research.

There is an urgent need to understand the physical nature of such a residue as it may play a key role [23] in chemical evolution. Residue from hydrocarbon ice irradiation was reported [24] to be fluffy and porous due to polymerization on carbon-rich ices [25] leading to micrometer-sized polymer layers [26]. In addition, the residues obtained by irradiating an aromatic (ben-
zene) molecular ice were found to contain geometric shapes such as micron sized cubes and spheres, sur-
rounded by nanoparticles [27]. The chemical analysis of the aromatic residues contained aromatic derivatives, and from biphenyls to polyphenyls [28], characteristic spectral signatures of aromatic derivatives were present in the vacuum ultraviolet (VUV) photoabsorption spec-
trum of the residue [27]. Two possibilities for the com-
position of the residues can be discussed, (i) mixed aromatic/aliphatic organic nanoparticles (MAONs) as dust in the place of (ii) polycyclic aromatic hydrocar-
bons (PAH), based on space-based observations in the infrared [29, 30]. So there is a dire need for more labor-
atory experiments to understand the physico-chemical nature of the residue from astrochemical ice irradiation.

The report of the possible presence of C_{24}, a small graphene sheet [31, 32], has led to estimates of the concentrations of graphene in different regions of the ISM [33] and suggests the presence of larger graphene sheets. Furthermore, it is expected that the presence of graphene may be concomitant with the presence of the Buckminster fullerene, C_{60}, and the larger fullerene, C_{70}, already identified in the ISM [34]. The formation of C_{60} is proposed to follow one of two chemical path-
ways; bottom-up [35, 36] and top-down [37] model. The top-down model starts with PAH being converted to a larger graphene sheet which eventually synthesizes C_{60} following the pathway suggested by Berne and Tielens [38]. Therefore, the presence of C_{60} can be used as a tracer to look for graphene-rich clouds in the ISM.

Hydrogen loss from MAONs may also form graphene. However, to-date, the formation of these allotropes of carbon has only been examined at higher temperatures that do not resemble the cold dust containing an icy mantle. Efforts were taken to understand the formation of carbon clusters in icy conditions, up to C_{20} were observed to form at low temperature in an irradiated methane in neon ice matrix [39]. The results reported suggest that carbon atoms combine to form chain of molecules, even at such lower temperatures, which if further pursued may lead to the formation of graphene.

The recent identification of molecules containing an aromatic ring with a side chain in the ISM, such as benzonitrile [40] that can readily form [41, 42] at ISM conditions, provides another possible route to the for-
mation of complex carbon structures such as graphene. Irradiation of such a compound may lead to dissociation liberating the aromatic ring which may then assemble with other rings to form a more complex PAH. In this paper, we report for the first time the results of photo-
processing of benzonitrile ice prepared under ISM con-
ditions.

2 The laboratory analogue experiment and observed carbonaceous residues

VUV photoprocessing of benzonitrile (C_{6}H_{5}CN) ices, sourced from Sigma Aldrich − purity ≥ 99%, was studied at two beamlines of Taiwan Light Source (TLS), TLS BL03 and TLS BL21A2, at the National Syn-
chrotron Radiation Center. The details of the experi-
mental system used can be found elsewhere so only salient details are presented here [39, 43, 44].

A benzonitrile ice film was formed at 4 K, by depositing benzonitrile at 5 × 10^{-7} Torr for 60 s (nearly 30 monolayers) onto Lithium Fluoride (LiF) and Potassium Bromide (KBr) substrates, before irra-
diation (Figure S1). The ice was then irradiated by 9 eV photons for nearly 9 h and subsequently observed using VUV and infrared (IR) spectroscopy (Figure S1 & S2). Post irradiation, the sample was warmed at the rate of 5 K min^{-1} where VUV and IR spectra were recorded at regular intervals. After attaining 300 K, the UHV chamber was decompressed to remove the substrate containing the residue (Figure S3) for ex situ investig-
ation using electron microscopy (Figure S4). The sub-
strates used were new, not used in any other experiment before, and were cleaned using alcohol before loading on to the substrate holder. Post irradiation and warmup to 300 K, the substrate collected from the substrate holder was then stored in a sterile window holder box.

The amount of sample deposited is quite high for the VUV spectroscopy; however, the aim of these ex-
periments to synthesize residue from benzonitrile irradiation so, in order to synthesize sufficient residue and to obtain a good VUV spectrum of the residue, the amount of sample used before irradiation resulted in a satu-
rated VUV photoabsorption spectrum of benzonitrile at lower wavelengths (110 nm–260 nm). However, in the higher wavelength region, 260 nm–290 nm, due to the lower absorption cross section three bands at 264.5 nm, 271.6 nm and 279 nm were observed. After irradiation, these band positions and shape remain unchanged; however, the increase in baseline to higher absorption was an indication of absorption from newly synthesized molecule/residue synthesized by irradiation.

Using IR spectroscopy, in addition to the character-
istic bands corresponding to the amorphous benzoniti-
trile ice, the appearance of a new band at 2133 cm^{-1}, that corresponds to the NC stretching [45], was clearly observed after irradiation. During warming of the ice, IR spectra revealed a phase change to create crystalline benzonitrile ice at 150 K. Further warming the ice to
room temperature resulted in desorption of the benzonitrile but left a residue on the substrate. Such a residue was not observed in a benzonitrile ice that was warmed from 4 K to room temperature without irradiation.

The residue spectrum had absorption bands in the IR (Fig. 1), quite similar to the benzonitrile characteristic absorption band suggesting the residue could be an aromatic derivative (containing nitrogen). In addition, the residue spectrum recorded in the VUV after warming the irradiated ice was also observed to contain the characteristic aromatic ring absorption at 190 nm and an extended absorption tail until 300 nm (Fig. 1). Such a spectrum in the VUV is characteristic of aromatic derivatives [27]. Thus, both spectroscopic techniques provide compelling evidence that the residue synthesized by VUV irradiation contains aromatic molecules. However, experimental evidence also suggests that the availability of carbon atoms in irradiated ices [46] may lead to the formation of carbon clusters or carbon only molecules [39].

The appearance of characteristic features corresponding to non-volatile carbon/carbon–nitrogen clusters was not observed in VUV and IR spectra, but this could be due to lower concentrations in the residue. Therefore, in order to investigate the synthesis of non-volatile carbon only or carbon–nitrogen clusters from the ice irradiation, the sample was imaged using High Resolution–Transmission Electron Microscopy (HR-TEM), for which the residue obtained was scratched on to a Quantifoil TEM grid. The sample transfer from the window to the TEM grid was performed using a restricted air flow sample preparation table to avoid contamination. The grid containing the sample was then subjected to Ar + O₂ plasma cleaning, to remove any macroscopic dust from the lab environment, before loading the sample for TEM imaging. The blank experiment (benzonitrile ice without irradiation) did not show any sign of residue present on the windows so the resulting TEM image from the blank experiment did not show any nano-sized particle.

In the images obtained clusters of aligned atoms arranged within the structure of a quantum dot, max length ~ 3 nm were observed. The d spacing [47] measurement suggests the observed structure could be a graphene quantum dot. Such dots were seen in many places in the residue (Fig. 2). Several nanosheets were also observed. Upon closer examination, on a scale of < 1 nm, this sheet was found to contain a hexagonal arrangement of atoms (Fig. 3), a structure that which confirmed by the electron diffraction data (Fig. 3). The energy-dispersive spectrum (EDS) showed carbon and nitrogen to be present in this sheet (Fig. 3). From these observations, we can conclude that within this residue, there is evidence for the presence of nitrogen-doped graphene (N-Graphene). In this case, the quantum dot and N-graphene sheet were part of the other components present in a residue synthesized in laboratory analogue simulating ISM cold dust condition. The other components will be discussed in a separate publication.

3 Discussion

A laboratory analogue experiment of the irradiation of an icy mantle made of the recently discovered benzonitrile molecule prepared under ISM conditions has revealed, for the first time, the formation of nanoscale structure analogous to a quantum dot and N-graphene sheet. This observation has important consequences for our understanding of the formation and abundance of PAH molecules in the ISM. Nevertheless, applicability to astrophysical scenarios needs further investigation which requires deducing the irradiation dosage, ice composition and competing processes in a heterogeneous ice.

A new and simple route to the formation of complex PAH structures is demonstrated by irradiation of a simple aromatic compound. The atoms of carbon and nitrogen released by photodissociation easily diffuse within the ice upon warming leading to the formation of nanostructures. The presence of graphene suggests that C₆₀ / C₁₀₇, via the top-down model, and carbon nanotubes, by rolling up the (graphene) sheet, may also form in irradiated aromatic ices. Graphene containing dust may be the source of C₂₄ reported by [31, 32]. A variety of hydrocarbons and hydrocarbys may result by etching graphene in hydrogen-rich environments commensurate to conditions in the ISM [48]. While graphene in hydrogen-rich environments can synthesize PAH molecules, the presence of N-graphene may synthesize polycyclic aromatic nitrogen heterocycles (PANHs), signatures of which may be present in spectral collection from the observations of different regions of the ISM clouds, using space- and ground-based observatories.

Titan’s stratospheric polar clouds also contain benzene ices [49]. The chemical composition of surface ices on Titan is known, from laboratory experiments [50], to harbour aromatic molecules such as benzene. Therefore, in a nitrogen-rich environment (such as Titan) where benzene ices are also present, benzonitrile may readily form [42], [51]. Our experimental result showing N-graphene synthesis from benzonitrile ices therefore suggests that N-graphene may also be a component of Titan’s icy clouds and surface ices. This prediction may be supported by CAPS ELS data collected during the T16 encounter of the Cassini spacecraft, at approximately 1000 km altitude [52], which reveals material with a few 1000’s of Dalton. These high masses have been attributed to the presence of tholins [53]; however, N-graphene might co-exist with tholins in the chemically complex clouds of Titan. Therefore, adding the possible presence of N-graphene (and even quantum dots) to atmospheric models of Titan may provide clues to those physico-chemical processes leading to formation of Titan’s haze.
Fig. 1 Spectra of the residue, after benzonitrile irradiation at 4 K and then warmed to room temperature, (left) in the VUV and (right) in IR wavelengths. The residue spectra were recorded at in situ UHV conditions before decompressing the chamber to remove the windows.

Fig. 2 HR-TEM image of the residue showing arrangements of atoms resembling a Quantum Dot.
4 Conclusion

Irradiation of a 4 K benzonitrile ice film by 9 eV photons and subsequent warming to room temperature has demonstrated the formation of nanostructures including quantum dots and N-graphene. This important observation shows that the bottom-up model to synthesize graphene is also feasible for a cold dust grain in the ISM, which can subsequently lead to the synthesis of larger PAH molecules in a hydrogen-rich environment. The search for graphene sheets in the ISM should therefore be made by looking at regions where benzonitrile has been identified. Astrochemical dust models should then consider the presence of graphene and how it may influence the productivity of PAH and other hydrocarbons.

In the solar system, since benzonitrile is a likely component of the rich PAHs inventory present on Titan, these results suggest that we can also expect the presence of N-graphene (and even quantum dots) on that moon. Cassini flyby data should therefore be reviewed to look for any spectral signatures of N-graphene and Titan atmosphere models developed to explore synthetic routes of production in the Titan haze.

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Author contribution

BS contributed to conceptualization, supervision, project administration, methodology, investigation, formal analysis, visualization, funding acquisition, and writing—original draft. KKR contributed to investigation, formal analysis, and visualization. MA contributed to investigation, formal analysis, and visualization. DS contributed to investigation
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