***Research Article***

Chemical Review and Letters
journal homepage: www.chemrevlett.com
ISSN (online): 2645-4947(print) 2676-7279

# The possibility of a two-step oxidation of the surface of $C_{20}$fullerene by a single molecule of nitric (V) acid, initiate by a rare [2+3] cycloaddition

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ARTICLE INFO

Received 24 January 2019
Received in revised form 14 February 2019
Accepted 20 February 2019
Available online 20 February 2019

*Keywords:*
nitric (V) acid
$C_{20}$ fullerene
molecular mechanism
PES
reaction channels

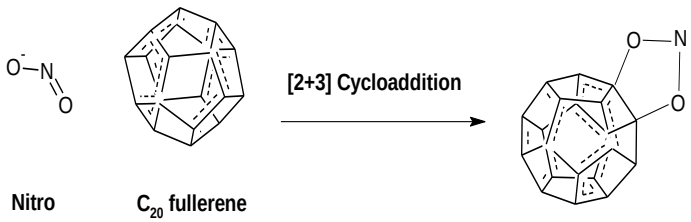
ABSTRACT

Oxidation of fullerenes, carbon nanotubes, and graphene, is one of the first proposed and successful approaches for further functionalization of these nano dimension carbon allotropes. Also, the C20 fullerene, as the smallest known carbon cage, is one of the most important species, in the future of nanotechnology. In this regard, the potential energy surface (PES) study suggests that reaction between nitric (V) acid and $C_{20}$ fullerene, first leads to the production of a relatively meta-stable kinetically allowed intermediate *via* a [2+3] cycloaddition. After the intermediate is produced, it would subsequently be decomposed to a $C_{20}O$ open-shell fullerene and a $HNO_2$molecule. Such oxidations were observed *via* the reaction between strong acids and some of the nano-sized carbon allotropes like carbon nanotube CNTs or spherical fullerenes. The results showed that the produced intermediate directly changes to the final product of oxidation, in a fast process.

## 1. Introduction

As generally known, the nitro group could exist in aliphatic nitro compounds [1], nitroarenes [2], and as part of nitric (V) acid molecule [3-6]. Many years ago, Huisgen was establish [7], that -N(O)O moiety have nature of 1,3-dipole, similarly as azomethine ylides, imine oxides and many others, which easily react with unsaturated systems according to [2,3] cycloaddition scheme [7-10]. In consequence, above compounds may potential, attractive build blocks for synthesis of 1,3-dioxazole derivatives via [2,3]- cycloaddition [8-10] with alkene/alkyne dipolarophiles. At this time no report on a product formed from [2,3]- cycloaddition involving nitric (V) acid[11,12]but instead, there are many reports revealed about oxidation of graphenes, nanotubes, and fullerene family by using this acid [13,14].There is a possibly that a meta-stable intermediate emerges between reactants and oxidation product.

A literature review revealed that, the C-C double bond of the $C_{20}$fullerene isone of the most kinetically suitable unsaturated species for the reaction with 1,3-dipoles. According to the recent reports, the $C_{20}$ fullerene could perform [2,3]- cycloaddition reaction with dipolar species in a very fast process without needing any catalyst or accelerating agents [15,16].Also a few reports revealed about adsorption of nitrogen dioxide on the surface of the $C_{20}$ fullerene [17]. Therefore, the $C_{20}$ fullerene was selected as an unsaturated species to be used in reaction with the nitro functional group (Scheme 1) to produce an intermediate which subsequently changes to an oxidation product.




**Scheme 1.** Chemical adsorption of nitro species on the surface of $C_{20}$ fullerene.

Nitric (V) acid is widely used in different types of chemical reaction and synthetic procedures [18-20]; thus, it could be a candidate as the selected species that carry the nitro functional group in reaction with $C_{20}$ fullerene. Due to the aforementioned factors, in this project, first, we devised a scheme containing the [2+3] reaction of the $C_{20}$ fullerene and nitric (V) acid (see Scheme 2) and

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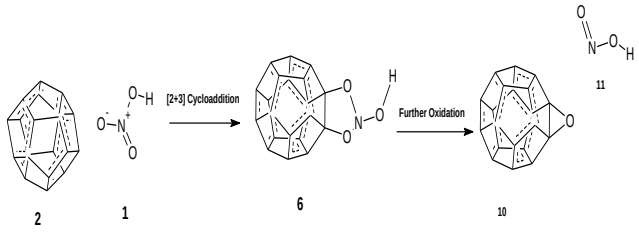
investigated the possibility as well as the kinetic and thermodynamic preference of this reaction, continued by further oxidation of the intermediate to make to final product of the reaction. Due to the semi-conductivity character of $C_{20}$ fullerene [20], the results of this project may be useful to introduce a way to create a new

angstrom sized semiconductor, via the mild oxidation of the $C_{20}$ fullerene.




**Scheme 2.**The reaction of nitric (V) acidwith $C_{20}$ fullerene and other olephines

## 2. Computational details

All the calculations were performed by using the Gaussian 03 chemical quantum package.[21] Geometries of critical structures were optimized by using density functional theory method at the B3LYP/6-311G(d,p) level of theory [22-24].The same functional has been recently used for explanation of mechanistic aspects of several [2+3] cycloadditions involving different components [25-27].The synchronous transit-guided quasi-Newton (STQN) method [28,29] was applied in order to find the transition state structures (TSs). To calculate the thermodynamic energy of each state, the frequencies of each species were taken.

The transition states were verified by using the vibrational analysis as well as via analysis of intrinsic reaction coordinates (IRC) [30,31]. The natural bond orbital (NBO) analysis [32,33] was applied to find the electrical charge of atoms in both reactant and transition states. The Pauling relation [34] was used to detect the related partial bond orders. Global Electron Density Transfer (GEDT) [35] was calculated by using the following relation;

$$GEDT=|\Sigma q_A|$$

where; $q_A$ is the net Mulliken charge; and, the sum includes all the atoms of dipolarophile species. To take the solvent into account, we performed our calculations using the conductor-like polarizable continuum model (CPCM) [36].

## 3. Results and discussion

Firstly, we shed light on the reaction course of [2+3] cycloaddition between nitric acid **1** and fullerene **2**. For this purpose, several structures were drawn as a default for each species to serve as input files and were then optimized to give stable or meta-stable geometries. Some valid geometries were found for the reactants, the product, the transition states and the meta-stable intermediate of the hypothetical route (Fig 1). Also, the Born-Oppenheimer Molecular Dynamics (BOMD) Simulations which would help to find some unidentified intermediates as well as other reaction species, were used in this investigation (Figure 2). The time of the simulations was 4700 femtoseconds.

Due to the latest reports about the possibility of stepwise routes in reaction pathways of [2+3] cycloaddition [37-40]. We considered and examined the stepwise routes. Following attempts, a meta-stable cyclic intermediate between reactants and product was detected. It was found that reaction **1**+**2** is initiated by the formation of C5-O3 and O1-C4 bonds as well as the cleavage of C4-C5 and N2-O1 π bonds.

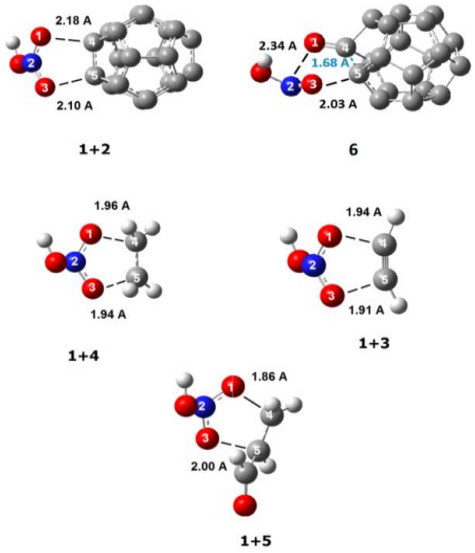



**Figure 1.** Views of transition states for [2+3] cycloaddition involving nitric (V) acid according to the B3LYP/6-311G(d,p) data.

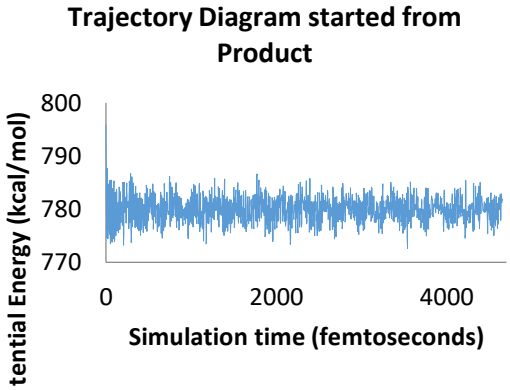



**Figure 2.**The BOMD simulation started from the product of the reaction of **1**+**2**. The simulation time was about 4700 femtoseconds.

In the TS of the first step of the reaction (between reactants and intermediate), the length of C5-O3 and O1-C4 bonds is2.11 and 2.18 angstrom; respectively. The

Pauling relation [34] was used to determine the related partial bond orders, and, 0.091, 0.072, 0.967, and 0.875 values were obtained for C5-O3, O1-C4, C4-C5 and N2-O1 bonds, respectively. The calculated partial bond orders indicated that for C5-O3 and O1-C4 only 9.1% and 7.2% values were formed, respectively. Whereas π bonds in C4-C5 and N2-O1 were broken to 3.3% and 12.5% respectively. The extent of the broken
and formed bonds in the TSs shows that a rather synchronous mechanism occurs for the [2+3] cycloaddition reaction of $C_{20}$fullerene **2** with nitric (V) acid **1**.For the TS of the second step of the oxidation (decomposition of the intermediate), the length of the C4-O1 bond become shorter (1.25Å) in order to form a C=O double bond. Along with formation of O1-C4 double bond, the lengths of N2-O1, C4-C5, and O3-C5 bonds were being longer to break those σ bonds (2.34 Å, 1.68 Å, and 2.03 Å, respectively).

**Table 1** Key parameters for structures of [2+3] cycloaddition reactions involving nitric (V) acid **1** in the gas phase

| Reaction | Structure | Interatomic distances [Å] | | NBO charges | | | | | GEDT [e] |
|---|---|---|---|---|---|---|---|---|---|
| | | O1-C4 | O3-C5 | O1 | N2 | O3 | C4 | C5 | |
| **1+2** | reactants | - | - | -0.347 | 0.753 | -0.408 | -0.387 | 0.138 | - |
| | TS | 2.18 | 2.10 | -0.367 | 0.668 | -0.329 | -0.026 | -0.004 | 0.11 |
| | **6** | 1.39 | 1.39 | -0.452 | 0.387 | -0.452 | 0.232 | 0.232 | - |
| **6** | TS | 1.25 | 2.03 | -0.697 | 0.399 | -0.352 | 0.340 | 0.141 | -0.412 |
| | 10 | 1.19 | - | -0.523 | -0.377 | -0.317 | 0.480 | -0.048 | - |
| **1+3** | reactants | - | - | -0.399 | 0.753 | -0.328 | -0.230 | -0.236 | - |
| | TS | 1.94 | 1.91 | -0.374 | 0.606 | -0.340 | -0.130 | -0.120 | 0.20 |
| | **7** | 1.39 | 1.39 | -0.394 | 0.398 | -0.394 | 0.021 | 0.021 | - |
| **1+4** | reactants | - | - | -0.393 | 0.751 | -0.329 | -0.377 | -0.377 | - |
| | TS | 1.96 | 1.94 | -0.380 | 0.590 | -0.347 | -0.273 | -0.270 | 0.26 |
| | **8** | 1.42 | 1.42 | -0.449 | 0.373 | -0.373 | -0.061 | -0.061 | - |
| **1+5** | reactants | - | - | -0.345 | 0.754 | -0.429 | -0.224 | -0.317 | - |
| | TS | 1.86 | 2.00 | -0.379 | 0.597 | -0.322 | -0.223 | -0.202 | 0.20 |
| | **9** | 1.41 | 1.42 | -0.434 | 0.382 | -0.469 | -0.052 | 0.008 | - |

Table 1 shows the charge distribution in the reactants, TSs, as well as the charge differentiation between TSs and reactants **1**+**2** by using the natural bond orbital(NBO) analysis. The results show that a small negative charge increased on C13, and O22 atoms at TS. But in the case of the second step oxidation transition state, the negative charges increase at N2 and C4. And the significant GEDT value (-0.412 eV) shows that the TS of the second step oxidation is relatively polar.

**Table 2** Activation parameters for the [2+3] cycloaddition reactions involving nitric (V) acid **1** in gas phase and different solvents (T=298.15K; $\Delta G^{0,\#}$ and $\Delta H^{0,\#}$, are in kcal $mol^{-1}$)

| Reaction | Environment | $\Delta G^{0,\#}$ | $\Delta H^{0,\#}$ |
|---|---|---|---|
| **1**+**2** | Gas phase | 17.40 | 14.87 |
| **1**+**2** | n-heptane | 18.93 | 14.75 |
| **1**+**2** | $CCl_4$ | 18.95 | 14.82 |
| **1**+**2** | Toluene | 18.96 | 14.86 |
| **1**+**2** | Acetonitrile | 19.44 | 15.29 |
| **1**+**2** | Water | 19.49 | 15.31 |
| **1+3** | Gas phase | 40.31 | 36.07 |
| **1+4** | Gas phase | 37.51 | 32.51 |
| **1+5** | Gas phase | 43.31 | 39.78 |

According to Table 2, the Gibbs free energy difference between reactants and the transition state was 17.40 kcal $mol^{-1}$ indicating that the first step of the reaction is possible process, at least, in the gas phase. When the impact of the solvent was examined, it was shown that due to the polarity of the solvent, the $\Delta G^{0,\#}$value is slightly increased and the reaction rate is decreased approximately. Changing the solvent from the n-heptane to toluene, acetonitrile, and water showed that the reaction rate is decreased slightly by increasing the *polarity.*

It might be due to low polar nature of the transition state. Table 1shows that the GEDT value for the TS is approximately 0.11e. Such a small value of GEDT confirms the rather weak-polar character of the transition state for the first step cycloaddition.

Next, we analyzed similar reactions involving other dipolarophiles (Scheme 2). It was found that, these processes proceed via TSs with similar synchronicity (Table 1, Fig. 1). However, their polarities are significantly higher than in the case of cycloadditions between nitric (V) acid (**1**) and $C_{20}$ fullerene (**2**). Subsequently, energetic aspects of these reactions were

examined.

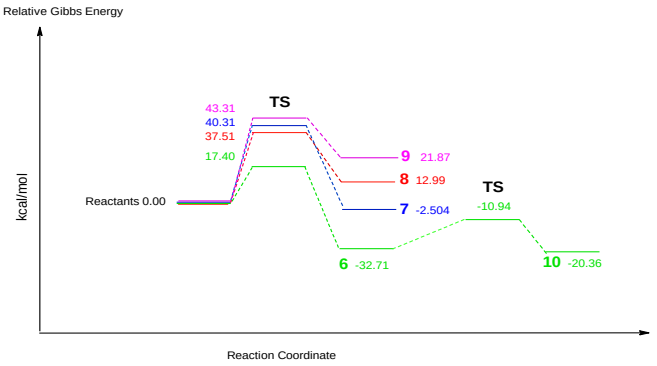



**Figure 3.** The relative Gibbs free energy diagram for the purposed reactions

According to Table 2, the [2+3] cycloaddition between nitric (V) acid **1** and usual unsaturated compounds like ethyne **3**, ethylene**4**, and propenal**5** has a $\Delta G^{0,\#}$ of 37.51, 40.31, and 43.31 kcal $mol^{-1}$, respectively. It shows that in non-catalyzed conditions, nitric (V) acid is unable to react with usual olephines during a proposed [2+3] cycloaddition. Thus; the [2+3] cycloaddition of nitric (V) acid **1** with $C_{20}$ fullerene **2** with a $\Delta G^{0,\#}$ of 17.40 kcal $mol^{-1}$, is noteworthy in this case. In Figure 3, the relative Gibbs free energy of the reactants for each route was relatively supposed 0.00 kcal $mol^{-1}$.The results of the Gibbs free energy surface for the reaction pathways confirm that the [2+3] reaction of nitric (V) acid and $C_{20}$ fullerene to give the meta-stable intermediate is both kinetically and thermodynamically possible. Also the intermediate undergoes a relatively fast, irreversible decomposition due to the $\Delta G^{0,\#}$of the second step reaction (21.8 kcal $mol^{-1}$).

## 4. Conclusion

Examining some of the usual unsaturated carbon bonds showed that in most cases, the nitro group (at least in the case of nitric (V) acid) was not intended to produce a [2+3] cycloaddition with those dipolarophiles. However; there is a possibility that a meta-stable intermediate emerges by reaction of the $C_{20}$ fullerene and nitric (V) acid. In this case, when we put $C_{20}$ fullerene as a dipolarophile, emergence of an intermediate, appears to be kinetically possible. Subsequently, the intermediate decomposes to produce a ketone form oxidized fullerene $C_{20}O$, and a $HNO_2$ acid. This result suggests two points; first, researchers might be able to simply oxidize $C_{20}$ fullerene by using nitric (V) acid; second, there is a concern that using nitric (V) acid in a reaction environment could poison and disable the $C_{20}$ fullerene as well. Moreover, the results of the solvent effect calculations showed that use of solvents could slightly decrease the rate of this process. Finally, the results of this project may be useful to introduce a way to create a new angstrom sized semiconductor, via the mild oxidation of the $C_{20}$ fullerene.

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## How to Cite This Article

Seyyed Amir Siadati; Karolina Kula; Esmaiel Babanezhad. "The possibility of a two-step oxidation of the surface of C20 fullerene by a single molecule of nitric (V) acid". Chemical Review and Letters, 2, 1, 2019, 2-6