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Published in:

Proceedings of the 9th International Conference on Theoretical and Applied Nanoscience and Nanotechnology, TANN 2025

DOI:

[10.11159/tann25.122](https://doi.org/10.11159/tann25.122)

Publication date:

2025

Document Version

Publisher's PDF, also known as Version of record

[Link to publication](#)

Citation for pulished version (HARVARD):

Belkhiri, S, Karim, BM, Meriem, FA, Sabri, T, Fouzi, TA & Mekhalif, Z 2025, NO_x Entrapping Onto Hybrid Ionic Liquid-Nanocrystalline NaY and Barium Oxide Decorated Carbon Nanotubes: Application To Nitrocellulose Stability. in J Zhang, A Kiani & M Rahmani (eds), *Proceedings of the 9th International Conference on Theoretical and Applied Nanoscience and Nanotechnology, TANN 2025*. Proceedings of the International Conference of Theoretical and Applied Nanoscience and Nanotechnology, Avestia Publishing, 9th International Conference on Theoretical and Applied Nanoscience and Nanotechnology, TANN 2025, Toronto, Canada, 10/06/25.
<https://doi.org/10.11159/tann25.122>

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NO_x Entrapping Onto Hybrid Ionic Liquid-Nanocrystalline NaY and Barium Oxide Decorated Carbon Nanotubes: Application To Nitrocellulose Stability

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Abstract -Although the high toxicity of DPA; it is still considered as one of the most used and efficient stabilizer of nitrocellulose (NC). In order to enhance the stability of NC, DPA is substituted partially by a green hybrid stabilizer; NaY nanocrystalline zeolite-BaO-MWCNTs-ionic liquid (IL). A comparative study between NC stabilized using diphenylamine (DPA) and NC stabilized using hybrid stabilizer is conducted in this study. MWCNTs are first functionalized with monocarboxylic aryl diazonium salt generated in situ, then BaO nanoparticles are formed using barium acetate. The properties of the resulting materials are investigated using different characterization techniques (TEM XPS, and PXRD). Nanocrystalline zeolite NaY is then synthesized without template under hydrothermal conditions through crystallization of gel in mesoporous system of functionalized multi-wall carbon nanotubes with BaO nanoparticles (BaO-MWCNT) with an internal diameter of 5~20 nm. The hybrid stabilizer (NaY-BaO-MWCNTs-IL) is prepared by physical mixing of BaO-MWCNT with ionic liquid, then incorporated to nitrocellulose and characterized using X-ray analysis (XRD). The crystallinity and the chemical stability of both nitrocellulose samples is studied using XRD, and Vacuum stability test (VST) respectively. The effect of aging on the mentioned properties is also conducted, where the NC samples have been subjected to an accelerated aging during 28 days at 80 °C. The obtained results using XRD, XPS, and VST techniques proved the compatibility between the NC and the prepared tertiary nanomaterial. Furthermore, VST analysis showed that the NC stabilized using DPA and NaY-BaO-MWCNTs-IL has lower quantity of evolved gas comparing to the NC stabilized using DPA alone.

Keywords: Nitrocellulose, Carbon nanotubes, Functionalization, Nanocrystalline NaY, Accelerated aging, Chemical stability.

1. Introduction

Nitrocellulose (NC) is a highly energetic material derived from cellulose, and it is widely used in various applications especially in propellants and explosives due to its excellent combustion properties. However, NC is susceptible to environmental factors, including moisture and temperature, which can significantly impact its stability and ballistic performance. This behaviour is directly related to NC nitrate groups, which are well known by their chemical instability and their autocatalytic decomposition even at room temperature. Generally, the nitrate ester decomposes through both hemolytic cleavage of O-NO₂ bond due to their low bonding energy (155 kJ/mol) and hydrolysis. The products obtained during these reactions (acid, nitrogen oxide and other radicals) act as catalyst for both hemolytic and hydrolysis decomposition (autocatalytic decomposition) [1-4]. These products affect significantly the stability of the NC based energetic materials, consequently, their safe handling and storage service life.

Stopping the decomposition of the nitrate ester is not yet discovered but decreasing the autocatalytic decomposition can be obtained by using different type of additives called stabilizers. These stabilizers will attract and interact with the free radicals formed and therefore decrease the autocatalytic reaction. The mechanism of action of these stabilizers can be

by several way i) by attraction and fixation of the NO_x , ii) by eliminating and absorbing the water; iii) by neutralizing the acid formed. The products obtained throw the interaction between the stabilizer and radicals are also considered as stabilizers and they are named daughter products [5-15].

Those, a serious problem of inflammation or detonation depending on the degree of the confinement [1]. Efforts to enhance the stability of NC have led to the development of various techniques, including the use of stabilizers [12-15].

One criterion that must be presented in all kind of stabilizers is the compatibility of these materials with the nitrate ester formulation. The stabilizers that were used are generally aromatic amine and urea derivate elements including diphenylamine (DPA), and centralite, which are the most used stabilizer in homogenous propellants formulations. These stabilizers act generally by absorbing and trapping the generated element during the thermal decomposition and therefore slowing the autocatalytic decomposition [16-19]

Recently ionic liquids are considered as the next generation material that are widely used in different field due to their unique characteristic and properties, Ionic liquids (ILs) are molten salts with a melting point below 100 °C. ILs allows the reaction to be carried out at temperatures sometimes reaching 450 °C without significant degradation unlike organic solvents. The wide use of IL in catalysts is related to their ability to dissolve various inorganic and organometallic compounds. In this way, the catalyst can be fixed in the medium by grafting onto cations or anions or by trapping in a layer of the IL [20-21].

Nitrocellulose the most well-known type of nitrate ester and the main ingredient in the composition of all homogenous propellants; present the same stability issue as the other types of nitrate ester. Different studies have been conducted in the past in order to investigate the stability of NC using the common stabilizer DPA. Although its toxicity the military ammunition still can't substitute it by other stabilizer due to its high efficiency and compatibility with nitrocellulose [22-23].

In this investigation, the effect of NaY nanocrystalline zeolite-BaO-MWCNTs-ionic liquid on the chemical stability of NC is highlighted by XRD and VST techniques. The effect of accelerated aging up to 28 days on the properties of both stabilized nitrocellulose samples (DPA-NC and DPA-NC/BaO-MWCNTs-IL) is also studied.

2. Experimental Section

2.1 Chemicals and reagents

Choline chloride (ChCl , 99 %, Amresco), urea (99.5 %, VWR International), NaOH (98%, Acros Organics), acetone (99%, Chem-Lab), 4-amino benzoic acid (99%, ABCR GmbH & CO.KG) , Sodium nitrite (99.2%, Fisher Scientific UK), perchloric acid (70%, Merck Eurolabnv/sa), pentane (99%, Lab-Scan Analytical Sciences), barium acetate (99%, Merck), K_2CO_3 (99%, Panreacchimica), dimethyl formamide (DMF, 99.9%, Fisher Chemical), colloidal Ludox (HS40), sodium aluminate (Al_2O_3 : 50-56 %, Na_2O : 40-45 %, $\text{Fe}_2\text{O}_3 \leq 0.05$ %), are purchased from Sigma-Aldrich and deionized water(Fisher Chemical) are used without further purification. MWCNTs (NC 7000) (>95%) having a length of several (0.1-10) μm with an average diameter of 10 nm are supplied by Nanocyl SA (Belgium).

2.2 Apparatus

The Tecnai 10 Philips microscope is used for transmission electron microscopy (TEM) analysis. By dispersing the material in ethanol and placing a drop of suspension on a carbon-coated copper grid, the examined samples are created. The TEM is set up with an accelerating voltage of 80 kV, a current of 5 A, and a spot size of 3.

XPS spectra are recorded on a Thermo Scientific K-Alpha spectrometer using monochromatized Al $\text{K}\alpha$ radiation (1486.6 eV), and the photoelectrons collected at 0° with respect to the surface normal are analysed using a hemispherical analyser. The major peak of core level spectra is calibrated with respect to C1 s level fixed at 284.6 eV. The spot size of the X-ray source on the sample is 200 μm , and the analyser is operated with a pass energy of 200 and 50 eV for survey and high-resolution core levels spectra, respectively. A PANalytical X'Pert PRO multi-purpose diffractometer with Cu $\text{K}\alpha_1$ radiation ($\lambda=1.5418$) is used to conduct XRD analysis on the various samples. Data is collected using an X'Celerator detector with a step size of 0.0080/2 and a counting time of 24.7650 s over an angular range of 15-85/2. The voltage is set to 45 kV, while the current is set at 40 mA. X'Pert High Score software is used for background subtraction and peak tuning.

2.3 Decoration of carbon nanotubes with BaO

The decoration of MWCNTs with barium oxide nanoparticles is performed under infrared irradiation via an in-situ produced aryl monocarboxylic salt. Then, the barium oxide nanoparticles are generated with barium acetate [26]. This method is described in detail in the work of Bhakta et al. [27]. The resulting product is calcined at 500 °C under argon gas for 2 hours, the decorated MWCNTs are referred BaO-MWCNTs.

2.4 Synthesis of the hybrid NaY-BaO-MWCNTs stabilizer

The synthesis of NaY zeolite is carried out using a molar composition of 7.9 Al₂O₃ : 46 SiO₂ : 10.16 Na₂O : 0.7 Fe₂O₃ : 886 H₂[28]. Additionally, during aging of the prepared feed stock gel, BaO-MWCNT is added, then the mixture is transferred Teflon-lined inner chamber for hydrothermal synthesis. The obtained NaY zeolite-BaO-MWCNTs is recovered by low-speed centrifugation, washed with deionized water until the pH reached 8, and dried in an oven at 80°C for 24 h. The hybrid stabilizer (nanocrystalline NaY zeolite-BaO-MWCNTs-ionic liquid) is prepared by physical mixing of 8 g of the ionic liquid (IL) with 2 g of the nanocrystalline NaY zeolite-BaO-MWCNTs at room temperature under ultrasonic bath until the formation of homogenous paste.

2.5 Preparation of inorganic stabilizer-nitrocellulose mixtures

Two mixtures of 3% stabilizers and 97% of nitrocellulose are prepared. The first one is a mixture of NC/DPA and the second one is a mixture of NC/NaY-BaO-MWCNT-IL in this mixture the masse ratio of DPA and NaY-BaO-MWCNT-IL is 1/1. The preparation of these mixtures requires prior drying of the NC and the stabilizers used to remove the moisture contained in them. In order to obtain a homogeneous mixture, dissolution of the components in acetone is necessary with stirring until complete dissolution, then these two mixtures are spread out on a glass flat surface for drying at room temperature. This will allow the acetone to evaporate, and therefore the formation of a film of NC/DPA and NC/NaY-BaO-MWCNT-IL. After drying at room temperature all samples are cut into thin strips. Placed in labelled vials and then stored in a desiccator. These samples are used to carry out the accelerated aging and the various characterizations.

2.6 Characterization of BaO-MWCNT and NaY-BaO-MWCNT

2.6.1 Characterization by XRD

Figure 2 shows the XRD patterns of MWCNTs and BaO-MWCNTs, diffractograms (a) and (b) respectively, the appearance of others peaks at $2\theta = 21.43^\circ$, $2\theta = 36.19^\circ$, $2\theta = 41.62^\circ$, $2\theta = 57.48^\circ$ and $2\theta = 79.42^\circ$ is noticed. These peaks are similar to those reported in the literature [31], indicating that the nanoparticles formed are indeed barium oxide nanoparticles, demonstrating the efficiency of the followed approach.

The XRD pattern of the synthesized nanocrystalline NaY-BaO-MWCNTs is highlighted in Figure 2 (c). This diffractogram contains all the characteristic peaks of the nanocrystalline NaY zeolite (ICDD: 01-073-9997) which appear at $2\theta = 6.093$, 10.0502 , 11.7911 , 15.5549 , 20.2614 , 23.5610 , 26.9179 and 31.1642° corresponding to the crystal plan of (111), (220), (311), (222), (331), (440), (533) and (642) respectively. Furthermore, all the diffractions peaks of BaO-MWCNTs are observed.

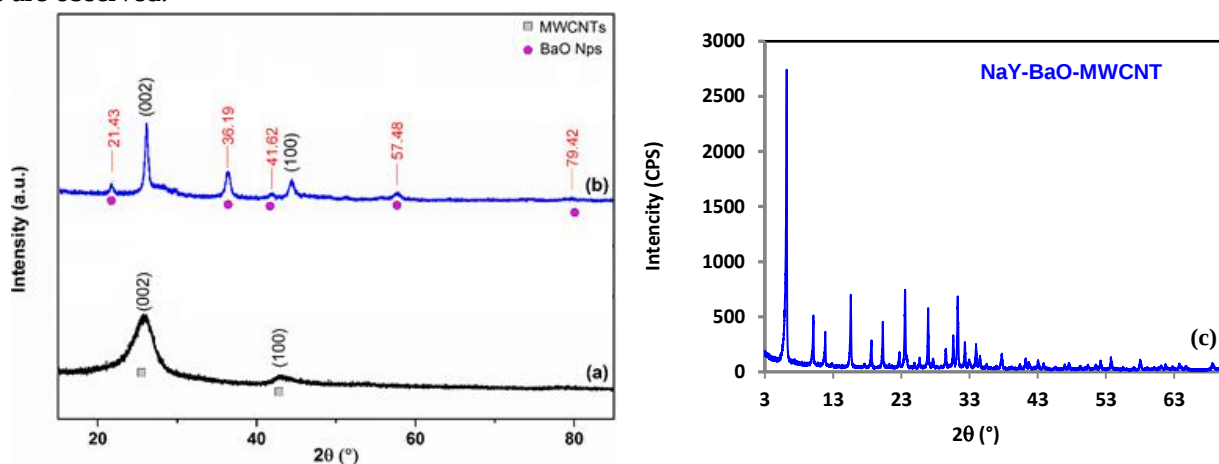


Fig. 2: X-ray diffractograms of: (a) MWCNTs and (b) BaO-MWCNTs, and (c) Nanocrystalline NaY-BaO-MWCNTs.

2.6.2 Characterization by TEM

Figure 3 shows the morphology of MWCNTs, BaO-MWCNTs and nanocrystalline zeolite NaY-BaO-MWCNTs. According to TEM image of MWCNTs, the nanotubes preserve their integrity after purification (Figure 3a).

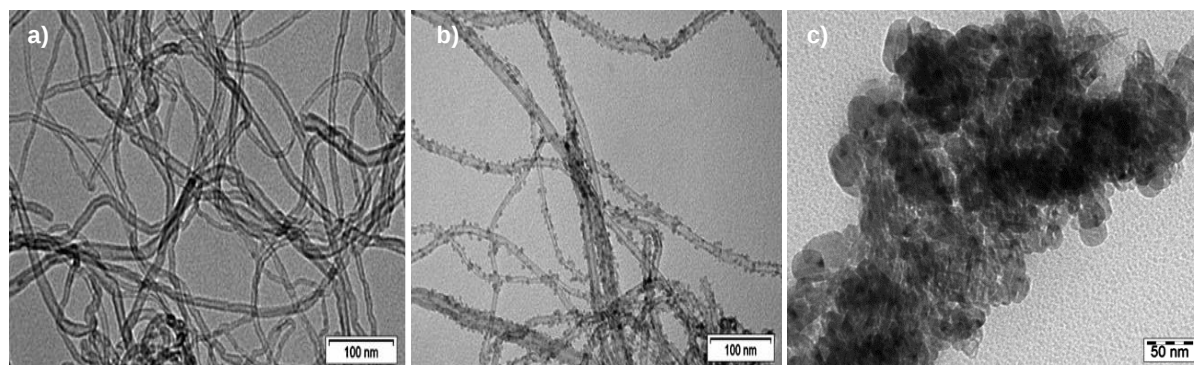


Fig. 3: TEM image of: (a) MWCNTs, (b) BaO-MWCNTs and (c) NaY-BaO-MWCNTs.

TEM image of BaO-MWCNTs (Figure 3b) shows a good distribution of BaO nanoparticles, which validates the efficiency of the method used for grafting these nanoparticles on the surface of MWCNTs [32]. Figure 3c shows nanocrystalline morphology of NaY-BaO-MWCNTs zeolite, with a small average crystal of about 40 nm.

2.7 Comparative study based on the crystallinity, and chemical stability during accelerated aging

Accelerated aging is becoming the most used method in order to evaluate the different properties of nitrocellulose based energetic materials in a short time interval at elevated temperature. In our study accelerated aging is performed using a thermostat block set at a temperature of 80 °C (± 0.1 °C) for a period of 28 days. The artificial aging is performed in order to establish a comparative study based on the crystallinity, the thermal decomposition behavior and the chemical stability, of the NC stabilized using DPA and the NC stabilized using a mixture of DPA and the NaY-BaO-MWCNT-IL nanocomposite

2.7.1 Crystallinity Study

XRD is used to investigate the compatibility of the synthesized green nano-stabilizer and the effect of accelerated aging on the crystallinity of NC-DPA. As shown in Figure 4 before accelerated aging NC-DPA and NC-DPA/NaY-BaO-MWCNT-IL presents the same characteristic peaks of pure NC, where a sharp pick at $2\theta=12.37^\circ$ and a broad pick and $2\theta=21.04^\circ$ are seen in the two XRD patterns. These two peaks are attributed to the superposing double peak assigned to one part of crystalline domain of NC and to the less ordered region of the nitrocellulose respectively [33-35]. These results indicate also that the presence of the prepared nano-stabilizer (NaY-BaO-MWCNT-IL) didn't affect the crystalline structure of the NC.

After 28 days of accelerated aging, the NC stabilized using the mixture of DPA-NaY-BaO-MWCNT-IL conserve almost its crystalline structure. However, for the NC stabilized with DPA alone show an apparition of another sharp pic at $2\theta=24^\circ$ confirming an increase of its crystallinity. The obtained percentages of crystallinity are evaluated using the Pearson VII method. Indeed, the increase in the crystallinity value of the NC-DPA after accelerated aging is due to its chemical degradation and the release of the NO_2 gazes. Hence, NaY-BaO-MWCNT-IL is more efficient stabilizer for NC under accelerated aging than the DPA alone.

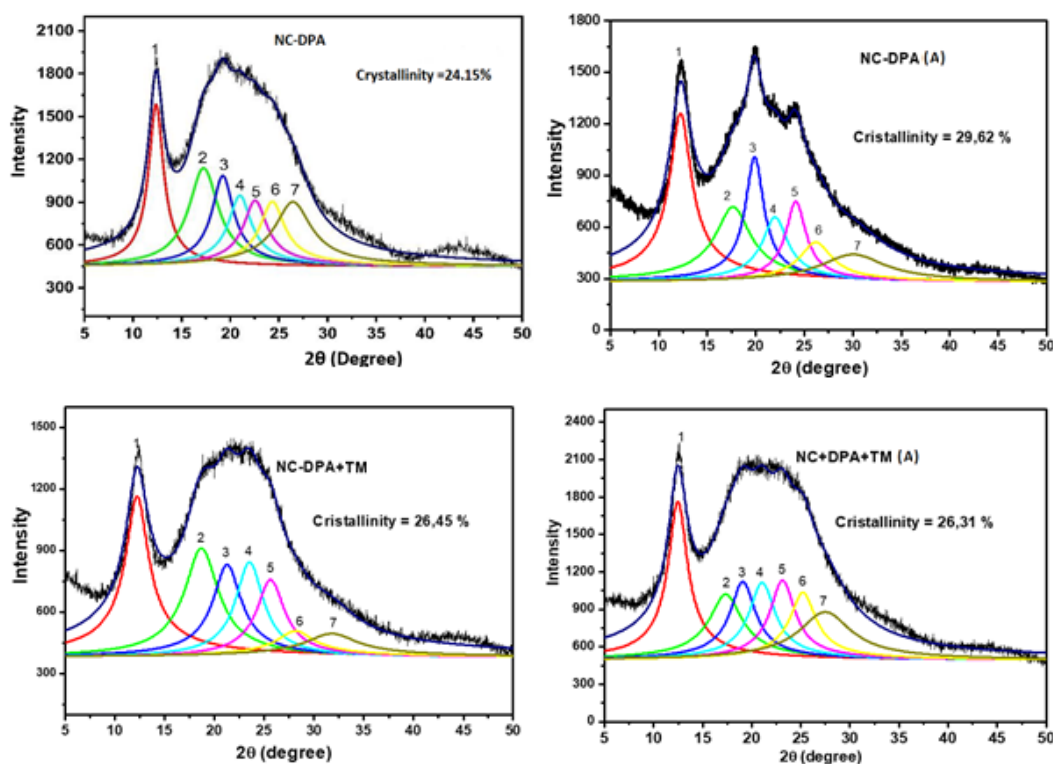


Fig.4: Peak separation of XRD profiles of NC-DPA and NC-DPA/BaO-MWCNTs-IL before and after accelerated aging (A, 28 days).

2.7.2 XPS results

Percentage compositions of the stabilized NC, obtained by XPS characterization are displayed in Table 1.

Table 1: Atomic composition of NC-DPA and NC-DPA/BaO-MWCNTs-IL before and after accelerated aging.

Atomic composition (%)	% C	% O	% N	% Si	% Na	% Al	% Ba	% Cl
NC-DPA	31.9	53.5	14.7	-	-	-	-	-
NC-DPA/BaO-MWCNTs-IL	35.1	51.2	10.6	1.70	0.70	0.70	0.023	0.042
NC-DPA (aged 28 days)	53.3	33.1	13.6	-	-	-	-	-
NC-DPA/BaO-MWCNTs-IL (aged 28 days)	37.4	49.64	9.8	1.70	0.71	0.69	0.022	0.041

These results show that NaY-BaO-MWCNTs-IL enhance NO_x retention by entrapping degradation gases through adsorption and catalytic interactions. This reduces nitrogen loss during thermal aging, mitigating autocatalytic decomposition. As a result, the overall thermal stability of nitrocellulose is significantly improved.

2.7.3 Chemical stability using VST

The chemical stability of the samples is performed using Vacuum stability test (VST). This test is a controlling, measuring, enabling evaluation of temperature stability from measurements of evolved gases from tested sample during long term isothermal heating. This test is performed according to STANAG 4556, where 1 g of the sample is heated at 100 °C for 40 h with pressure measurement reading each 1 min during the isothermal heating process [36-38].

The volume of gases given off by the samples is measured at the end of the isothermal heating period. This volume is calculated from the pressure values measured by the pressure transducers as a function of time. As the chemical stability is the most critic properties of nitrocellulose, the VST characterization is done every 7 days until reaching the 28 days for each sample. Table 2 represents the results of the VST test in term of evolved gas for the two mixtures (NC-DPA and NC-DPA/NaY-BaO-MWCNT-IL) during accelerated aging. From Table 2 several observation can be obtained i) the two samples of the NC stabilized by DPA and that stabilized by the mixture of DPA-NaY-BaO-MWCNT-IL release a volume of gases which increases as a function of the aging time ($V_{gas}(t=0d) < V_{gas}(t=7d) < V_{gas}(t=14d) < V_{gas}(t=21d) < V_{gas}(t=28d)$), which is an obvious result because the autocatalytic decomposition of NC which increase with time especially at elevated temperature. ii) The NC stabilized with DPA releases a volume of gas which increases linearly during the first week of aging, then it becomes constant until the second week. This volume evolves in the same trend during the third

week, and then it becomes stable until the fourth week of aging. However, the volume of gas evolved from the NC stabilized with DPA-NaY-BaO-MWCNT-IL remains stable in the first two weeks (no gases are detected), then it shows a significant increase from the third to the end of aging.iii). The quantity of the evolved gas of the NC stabilized with DPA alone is much higher than that stabilized with DPA/NaY-BaO-MWCNT-IL which show that added nano-stabilizer (NaY-BaO-MWCNT-IL) contribute to improve the chemical stability of NC by absorbing NO_x gases. These results highlight the stabilizing effect of NaY-BaO-MWCNT-IL (the ability to absorb and fix nitrogen oxides and other decomposition products) during the storage time. Nevertheless, the NC stabilised using DPA alone become instable from the first week of aging.

Table 2: Variation of evolved gazes by each mixture during the accelerated aging.

Composition	Volume of evolved gas (ml/g) at different periods (day (d))				
	0 d	7 d	14 d	21 d	28 d
NC-DPA	1.869	3.234	3.382	8.248	8.268
NC-DPA-TM	0.260	0.305	0.343	1.065	5.964

TM= tertiary mixture (NaY-BaO-MWCNT-IL)

3. Conclusions

BaO-MWCNT is firstly prepared using cost effective and efficient two steps method to impregnate barium nanoparticles on MWCNTs. Firstly, MWCNTs are functionalized with monocarboxylic aryl diazonium under IR irradiation, and then with barium (II) acetate as barium precursor by IR irradiation. Barium oxide nanoparticles are uniformly decorated onto MWCNTs, and their morphology and structure are investigated by TEM and XRD. The obtained BaO-MWCNTs is used as precursor during the NaY synthesis. The obtained nanocrystalline NaY combined to BaO-MWCNTs (NaY-BaO-MWCNTs) are characterized using several techniques. Later, NaY-BaO-MWCNTs is impregnated with ionic liquid and used as green tertiary organic-inorganic nano-stabilizer (NaY-BaO-MWCNTs-LI) to highlight its effect on the crystallinity and chemical stability of nitrocellulose under accelerated aging. The XRD study of the reference samples (NC-DPA and NC-DPA-NaY-BaO-MWCNTs) and the aged ones using the Pearson VII method show a higher crystallinity of NC-DPA compared to NC-DPA-NaY-BaO-MWCNTs. The green NaY-BaO-MWCNT-IL is more efficient stabilizer for NC under accelerated aging compared to DPA alone. The VST results showed that the NC stabilized using the mixture of DPA-NaY-BaO-MWCNT-IL highlight lower quantity of evolved gazes compared to the nitrocellulose stabilized with DPA alone. Indeed, the green NaY-BaO-MWCNT-IL stabilizer enhances the chemical stability of NC and can be considered as potential stabilizer of such ester nitric based energetic materials.

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