A simple and safe approach for simultaneous spectrophotometric determination of nitroglycerin and nitrocellulose in double base solid propellants

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Abstract
An accurate, simple and safe method was developed for simultaneous determination of nitroglycerine (NG) and nitrocellulose (NC) in double base solid propellants (DB propellants). The proposed method is based on alkaline hydrolysis of NG and NC, and followed by colored reaction of released nitrite ion with p-nitroaniline in the presence of diphenylamine in acidic media and produce azo dye. The absorbance of the azo dye was measured at 534 nm. Two sets of reaction conditions were developed. In the first set, at room temperature, only NG was hydrolyzed and calibration curve obtained. In the second set, at 60 °C, NG and NC were hydrolyzed simultaneously. Based on obtained amount for the NG at room temperature, and total amount of NG and NC at 60 °C, the amount of NC was determined by using stoichiometric equations. The calibration curve was linear over the concentration ranges of 0.2–5.0, 0.5–10 μg mL⁻¹ for NG and NC, respectively. The proposed method was successfully applied for the determination of NG and NC in DB propellants with good recoveries ranged from 99 to 101%, and RSD less than 2.0%. The method statistically compared based on t- and F-tests with those obtained in accordance to military standard method (MIL-STD-286). The results showed that the proposed method offers an accuracy and reliable approach for the determination of these compounds in DB propellants, and can be suggested as a routine method in military quality control laboratories.

Keywords Nitroglycerine · Nitrocellulose · Simultaneous Spectrophotometric determination · Double base solid propellants

Introduction
Double-base propellants (DB propellants) are best known as smokeless propellants and are used in guns and rockets. DB propellants consist mainly of nitroglycerine (NG) and nitrocellulose (NC) that used in rocket and gun applications as energetic base materials [1, 2]. The mechanical properties and shock sensitivities of these DB propellants are highly dependent on the mixture ratio of NC and NG [3–5]. In order to obtain superior mechanical properties, various additives are also included in the propellants to improve the mechanical or ballistic properties of the propellants. So according to the applications, solid propellants also included ballistic modifier or burning rate modifiers, stabilizers and plasticizers [6–8]. Due to high application and consumption of NG and NC in DB propellants, as well as needing to development of high-safety methods, there are always challenges for determination of these two compounds. The majority of reported methods in military standards like military standard 286 for the determination of NC in DB propellants are based on nitrometric method and extraction with toxic solvents. On the other hand, The significant drawbacks of reported method in military standard 286 for the determination of NG in DB propellants involves: the steps of methods are very long and time consuming, sample preparation is done in hard conditions with high toxic solvents and require complicated and expensive equipment such as gas chromatography and liquid chromatography. Therefore, the purpose of this study is to propose a safe, fast and user-friendly method for simultaneous determination of NG and NC in DB propellants without needing to expensive equipment.
Although, different analytical methods have been proposed for determination of NG and NC in different samples, such as, gas chromatography [9–11], liquid chromatography [12, 13] thermogravimetry [14, 15] spectrophotometric [16, 17], thin layer chromatography [18], ion chromatography [19].

Therefore, considering the important role of NG and NC in DB propellants, to date, no method has been reported in the literature and military standards such as Military Standard 286 for their simultaneous determination in DB propellants. Spectrophotometric methods are one of the greenest and attractive analytical methods because of availability of the instrumentation, speed and consumption of minimum solvents and instrumental hazards [20, 21]. On the other hand, according to the principles of green analytical chemistry about safety, reducing of time and using of toxic chemicals, development of safe and simple spectrophotometric method for simultaneous determination of NG and NC in DB propellants can be effective step towards the goals of green analytical chemistry [22]. The objective of this study is to evaluate the applicability of UV–Vis spectrophotometric technique as a safe, simple, selective, accurate, validated and user-friendly method for simultaneous determination of NG and NC based on differences in their hydrolysis rate within alkaline media. According to our research, this is the first report for simultaneous spectrophotometric determination of NG and NC in DB propellants.

Experimental section

Reagents

All chemical used in this study were analytical grade and used without any purification and double distilled water was used throughout the experiments. Para nitroaniline (99%), diphenylamine (≥ 98%), sulfuric acid, sodium hydroxide, ethanol (99.8%) and acetone was prepared from Merck. Nitroglycerine solution (1000 µg mL⁻¹), nitrocellulose and DB propellants were gift from Chemical Industries Department of Defense Industries/ Iran.

Stock nitrocellulose solution (1000 µg mL⁻¹) was prepared by dissolving an appropriate amount of nitrocellulose compound in acetone. p-Nitroaniline solution (0.2% w/v) and diphenylamine (0.2% w/v) were prepared in ethanol. A 5.0 M sulfuric acid solution was prepared by dissolving 27.8 mL of concentrated sulfuric acid in double distilled water and diluting to the mark in 100 mL volumetric flask. A 2.0 M sodium hydroxide solution was provided by dissolving 8 g of it in 100 mL double distilled water.

Apparatus

UV–Vis spectrophotometer Model 3310 from Hitachi Company (Japan) with 1 cm quartz cells was used for all the absorption measurements. All spectral measurements were performed using a blank solution as a reference. Magnetic stirrer (Hei Tec- Hidolph) was applied.

General experimental procedure

For determination of NG and NC in the DB propellants, two runs of experiments were performed for each sample. In the first step, appropriate volumes of NG and NC stock solutions (range from 0.2–5.0 µg mL⁻¹ for NG and 0.5–10 µg mL⁻¹ for NC) were transferred to a series 10 mL volumetric flasks and then 2 mL of 2 M sodium hydroxide was added and mix well in room temperature. Then 2 mL of 5 M sulfuric acid, 1 mL of p-Nitroaniline solution 0.2% (w/v) and 1 mL of diphenyl amine 0.2% (w/v) were added and mix well. The solution was diluted to the mark with water. Finally, the absorbance of each solution was measured at 534 nm against corresponding reagent blank. So, the equation of dependence of absorbance on the concentration of NG in room temperature was found to be as follow:

\[ A_1 = a_1 + b_1 C_{\text{NG}} \]  \hspace{1cm} (1)

That \( A_1 \) is absorbance of NG, \( a_1 \) and \( b_1 \) are intercept and slop of calibration curve respectively, and \( C_{\text{NG}} \) is concentration of NG. The second step was done as first one, expect after addition of 2 mL of 2 M sodium hydroxide to the test solutions, the solutions were placed in water bath at a controlled temperature of 60 °C for 6 min. So, the equation of dependence of absorbance on concentration of NG and NC at 60 °C during 6 min was found to be as follow:

\[ A_2 = a_1 + b_1 C_{\text{NG}} + a_2 + b_2 C_{\text{NC}} \]  \hspace{1cm} (2)

That \( A_2 \) is absorbance of total concentration of NG and NC, \( a_1 \) and \( b_1 \) are intercept and slop of calibration curve for NG respectively, and \( C_{\text{NG}} \) is concentration of NG, and \( a_2 \) and \( b_2 \) are intercept and slop of calibration curve for NC respectively, and \( C_{\text{NC}} \) is concentration of NC. The entire scheme of the procedure is shown in Scheme 1.

Sample preparation protocol of DB propellant for NG and NC determination

DB propellant was cut into short, thin slices and for each test 0.2 ± 0.1 g of sample, was weighed and dissolved in 20 mL of acetone at room temperature and the resulting solution was filtered for separation of insoluble particles.
The resulting solution was transferred into 100 mL volumetric flask and made up to volume with mixture of water/acetone (50:50) and used as a test solution.

**Results and discussion**

In general, NG and NC can be hydrolyzed in alkaline medium to produce nitrite ions [23–25]. On the other hand, produced nitrite ion can be reacted with p-nitroaniline (as a diazotizable aromatic amine) in the presence of diphenylamine (as a coupling agent) in acidic media to produce an azo dye as shown in scheme 2, which can be monitored spectrophotometrically by measuring of the absorption solution at 534 nm according to Fig. S1 [26, 27]. It was observed that the hydrolysis of NG in alkaline media immediately was completed after addition of sodium hydroxide solution at room temperature; while, hydrolysis of NC in the same conditions was relatively slow and its hydrolysis rates can be accelerated by increasing temperature and time. Because of these differences, NG and NC mixtures could be determined by selecting appropriate conditions.

Two runs were applied for each sample. For each run, all of conditions were the same except hydrolysis conditions with was variable for each run. For the first run hydrolysis was done in room temperature. At this temperature just NG was hydrolyzed after addition of sodium hydroxide solution. So, the obtained absorbance from reaction of nitrite ion with p-nitroaniline was proportional to concentration of NG. The temperature for the second run was 60 °C. At this temperature NG and NC can be hydrolyzed during 6 min after addition of sodium hydroxide solution. So, the obtained absorbance from reaction of nitrite ion with p-nitroaniline was proportional to concentration of NG and NC. Therefore, concentration of each analyte can be calculated by two simultaneous calibration equations.
Optimization of parameters affecting simultaneous determination of NG and NC

The experiments were carried out to develop a simple and fast method for simultaneous determination of NG and NC in DB propellants. So, in order to obtain a highly sensitive method for determination, some of the parameters that affect the process were examined within the appropriate range and their optimal values were found. Thus, to optimize the conditions for simultaneous determination of NG and NC two categories of parameters were considered: (i) a group that affects the efficiency of the hydrolysis process, and (ii) a group that affects the reaction of nitrite ion with p-nitroaniline and absorbance of system. So, in order to achieve full advantage of procedure the effect of important variables on performance and reaction conditions were studied and optimized.

Optimization of hydrolysis process

Since the hydrolysis of NG and NC occurs in an alkaline media, the concentration of sodium hydroxide has an important effect on the efficiency of hydrolysis. In order to find the optimal concentration of sodium hydroxide, the effect of this parameter was considered in the range of 0.1–0.6 mol L⁻¹. The results were revealed that the absorbance of system was increased up to 0.4 mol L⁻¹ (Fig. 1) and remained constant above this concentration, that revealed the hydrolysis process of NG and NC was completed in this concentration.

The effect of temperature on hydrolysis process was investigated in the range of 25–80 °C. The results of this evaluation were given in Fig. 2. According to the obtained results, increasing of temperature did not considerable change in NG absorbance, but in the case of NC, with increasing of temperature the absorbance of system was increased up to 60 °C and remained constant above it. So, it is concluded that hydrolysis process can be occur in room temperature for NG, but this process is very slow for NC in low temperatures.

The kinetic properties of proposed method were investigated in the range of 1–10 min. After the addition of sodium hydroxide, the intensity of color and absorbance of system was recorded as a function of time for NG and NC. The results (Fig. 3) revealed that the hydrolysis of NG occurred within a few second after the addition of sodium hydroxide and remained constant over the time but for NC, absorbance of system was increased to 6 min and remained nearly constant above this time that revealed, the hydrolysis of NC is a time-dependent process and the process is completed over the time, while in the case of NG this process is time independent.
Optimization of parameters affecting the reaction of nitrite with para nitro aniline

Colored reaction of nitrite with diazotizable aromatic amine in the presence of diphenylamine can be done in acidic media. Therefore, various acidic media was investigated and sulfuric acid was shown good results from others. On the other hand, the concentration of sulfuric acid has important effect on reaction condition, so, the effect of its concentration was considered in the range of 0.25–2.0 mol L⁻¹. The results revealed in Fig. 4 that the highest absorbance was obtained for NG and NC when acid concentration was 1.0 mol L⁻¹ and remained constant at higher concentrations. Thus, the reaction of nitrite ion with p-nitroaniline was completed in this concentration. Thus, a concentration of 1.0 mol L⁻¹ was selected for further experiments.

The concentration of p-nitroaniline as diazotizable aromatic amine is important for producing of colored azo product. So, the effect of p-nitroaniline concentration on formation of colored azo product was studied in the range 0.005–0.05% (w/v). As Fig. 5 displays, the absorbance of system was increased up to 0.02% (w/v) and remained nearly constant above this concentration. That shows the reaction between p-nitro aniline and nitrite was complete in this concentration.

The effect of concentration of diphenylamine as a coupling agent was considered in the range of 0.005–0.04% (w/v). The results in Fig. 6 revealed that the absorbance of the system increased by increasing the diphenylamine concentration up to 0.02% (w/v) and it decreased at above that. So, 0.02% (w/v) diphenylamine was used as optimal concentration for further investigation.

Analytical performance and method validation

In this work, we studied the hydrolysis of NG and NC with time and reaction of nitrite from hydrolysis process with p-nitroaniline for simultaneous determination of NG and NC based on spectrophotometric method, which the analytical features of the optimized method are including regression equation, linear range, limit of detection and limit of quantification, repeatability. The calibration curves were constructed by using 10 concentrations of each analyte that subjected with proposed methods and it was concluded that the methods to be linear in the range 0.2–5.0 µg mL⁻¹ and 0.5–10.0 µg mL⁻¹ with a regression coefficient (r) of 0.9997 and 0.9996 for NG and NC, respectively (Fig. S2 and Fig. S3). The limits of detection (S/N = 3) were 0.1 µg mL⁻¹ and 0.25 µg mL⁻¹ respectively for NG and NC and on the basis

Fig. 4 Effect of concentration of sulfuric acid: conditions: concentration of NG: 1.5 µg mL⁻¹, concentration of NC: 5.0 µg mL⁻¹, concentration of sodium hydroxide: 0.4 mol L⁻¹, temperature: 60 °C, time: 6 min, concentration of p-nitroaniline: 0.02% (m/v), concentration of diphenylamine: 0.02% (m/v)

Fig. 5 Effect of concentration of p-nitroaniline, conditions: concentration of NG: 1.5 µg mL⁻¹, concentration of NC: 5.0 µg mL⁻¹, concentration of sodium hydroxide: 0.4 mol L⁻¹, temperature: 60 °C, time: 6 min, concentration of sulfuric acid: 1.0 mol L⁻¹, concentration of diphenylamine: 0.02% (m/v)

Fig. 6 Effect of concentration of diphenylamine, conditions: concentration of NG: 1.5 µg mL⁻¹, concentration of NC: 5.0 µg mL⁻¹, concentration of sodium hydroxide: 0.4 mol L⁻¹, temperature: 60 °C, time: 6 min, concentration of sulfuric acid: 1.0 mol L⁻¹, concentration of p-nitroaniline: 0.02% (m/v)
of \((S/N = 10)\), the LOQ values of proposed methods were 0.33 \(\mu g\) mL\(^{-1}\) and 0.83 \(\mu g\) mL\(^{-1}\) respectively for NG and NC. The figures of merit of this study were summarized in Table 1. It confirm the proposed methods have a high sensitivity for determination of NG and NC.

According to Table 1 under optimum conditions for each calibration set, following equations can be used for simultaneous determination of NG and NC in the mixtures.

\[
A_1 = 0.0914 + 0.3023C_{NG} \quad (3)
\]

\[
A_2 = 0.1529 + 0.3023C_{NG} + 0.1247C_{NC} \quad (4)
\]

which \(A_1\) and \(A_2\) are absorbance in first and second runs, \(C_{NG}\) and \(C_{NC}\) are concentration of nitroglycerine and nitrocellulose, respectively.

To check the precision of the method seven replicate experiments were performed and the relative standard deviation (RSD) were obtained for mixtures. The results are given in Table S1. As the results show, the RSD less than 2.0% that revealed the method has good repeatability and precision and observed the proposed method can be very excellent for the simultaneous determination of NG and NC in the binary mixtures and in the presence each other.

**Determination of NG and NC in synthetic mixtures**

In order to consider the accuracy and reliability of developed method, various synthetic mixtures of NG and NC were prepared and examined by proposed method. So, a solution of 100 \(\mu g\) mL\(^{-1}\) of each component was prepared, then different ratios were prepared according to Table 2 and investigated by the proposed method. As the results revealed, the relative errors for all measurements were in the range—1–4.0. The maximum relative errors for NG and NC were 4.0% and 2.0%, respectively.

**Selectivity**

A prominent feature of the proposed analytical method is the relative freedom of the method from the effect of interferences. To study the selectivity of the developed method, the influence of different ions on the determination of a mixture 3.0 \(\mu g\) mL\(^{-1}\) of NG and 2.0 \(\mu g\) mL\(^{-1}\) of NC was investigated under the optimum conditions. The tolerance limit was defined as the concentration of added ion that caused less than \(\pm 5\%\) relative errors. The results (Table 3) indicated that most of species in tolerance limit \((w\text{ species}/w\text{ analytes}) 200:1\) did not interfere in the determination in simultaneous determination of NG and NC. Therefore, it is concluded that the proposed method provides a method with high selectivity and without any interfering effect for simultaneous determination of NG and NC.

**Application of the proposed method for simultaneous determination of NG and NC in DB propellants**

In order to verification of the proposed method in DB propellant analysis, determination of NG and NC in DB propellants also was done by a standard method (Military Standard 286C). The obtained results from \(F\)-test and \(t\)-test at 95% confidence level (Table 4), revealed that there weren’t significance differences between the obtained results for developed method and standard method.

Applicability and reliability of proposed method and also matrix effects in determination of NG and NC in DB propellants was investigated under optimum conditions. Samples were prepared according to the sample preparation section that mentioned above and subjected with developed method. The obtained results are summarized in Table 5. As the results show, excellent recoveries ranging from 99 to 101% were obtained. the amounts of recoveries proved that the developed method was not sensitive to the effect of matrix and the method is applicable in complex formulation of DB propellants.

| Table 1 Analytical characteristics of the optimized method | Analyte | Calibration equation | Regression coefficient \((r^2)\) | Linear range/\(\mu g\) mL\(^{-1}\) | LOD/\(\mu g\) mL\(^{-1}\) | LOQ/\(\mu g\) mL\(^{-1}\) |
|----------------------------------------------------------|---------|----------------------|-----------------|------------------------|----------------|----------------|
| NG
| A = 0.302 C + 0.0914
| 0.9997
| 0.2–5.0
| 0.1
| 0.33 |
| NC
| A = 0.125 C + 0.0615
| 0.9996
| 0.5–10
| 0.25
| 0.83 |

| Table 2 Determination of NG and NC in laboratory prepared mixtures by the proposed method | NG/\(\mu g\) mL\(^{-1}\) | Relative error, % | NC/\(\mu g\) mL\(^{-1}\) | Relative error, % |
|------------------------------------------------|-----------------|-----------------|-----------------|-----------------|
| Taken | Found | Taken | Found |
| 0.25 | 0.26 | +4.0 | 0.5 | 0.51 | +2.0 |
| 0.5 | 0.49 | −2.0 | 1.0 | 0.98 | −2.0 |
| 1.0 | 1.01 | +1.0 | 2.0 | 2.02 | +1.0 |
| 2.0 | 1.98 | −1.0 | 3.0 | 3.05 | +1.066 |
| 5.0 | 5.1 | +2.0 | 2.5 | 2.45 | −2.0 |
| 3.0 | 2.97 | −1.0 | 1.5 | 1.54 | +2.6 |
| 4.5 | 4.6 | +2.22 | 3.0 | 2.97 | −1.0 |
| 2.0 | 1.95 | −2.5 | 1.0 | 1.02 | +2.0 |
Conclusion

Our investigation demonstrated that spectrophotometric determination method as a simple, sensitive, fast and user friendly and without interfering of matrix effects or separation steps can be successfully applied for quantitation of mixture of NG and NC in DB propellants with wide dynamic range and good accuracy, precision and sensitivity. In this study acetone solvent was selected as a suitable solvent for this purpose and hydrolysis and conversion to nitrite ion occurs in an alkaline media and acetone acts only as a solvent for sample preparation that this is one of the prominent advantages of this method, which can be done by selecting a suitable organic solvent for sample dissolution, hydrolysis reaction and measurement can be done in aqueous medium. So, in comparison with reported methods in military standard 286, this method is routine and low instrumentation and operation cost. To the best our knowledge, this is the first report about simultaneous determination NG and NC in DB propellants and due to the fact that so far no method has been reported in military standards for simultaneous determination of NG and NC in DB propellants, therefore, this report can be proposed as a reliable method with high accuracy in military standards such as military standard 286 (MIL-STD-286) as a first report for simultaneous determination of NG and NC in DB propellant samples. Finally, development of UV–Vis spectrophotometric method, provide rapid and low-cost instrumentation and operation procedure for simultaneous determination of NG and NC without needing to sophisticated instruments such as HPLC. So, the proposed method could be applied in quality control experiments for routine analysis of NG and NC in DB propellants and could also be extended for further studies in this field.

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Declarations

Conflict of interests The authors declare no competing financial interest.
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