



Development of an eco-friendly method to convert life expired composite propellant into liquid fertilizer

Mehilal*, K.I. Dhabbe, Anjali Kumari, V. Manoj, P.P. Singh, B. Bhattacharya

High Energy Materials Research Laboratory, Sutarwadi, Pune 411021, India

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ABSTRACT

Large quantity of composite propellants is produced as waste due to life expiry of missiles/rejection of propellant lots during manufacturing. The environmental protection agency does not allow the hazardous materials for open burning/open detonation. Therefore, a systematic study has been carried out to develop a method for the disposal of composite propellant into liquid fertilizer without affecting the environment. In this study, propellant compositions were digested in dilute nitric acid followed by neutralization with 5 M KOH solution to get precipitated out aluminium as aluminium hydroxide and finally the obtained liquid was treated with orthophosphoric acid for further neutralization. The liquid fertilizer, thus, obtained was characterized for nitrate and phosphate content using ion chromatography while ICP-AES was used for the estimation of potassium, aluminium and other noxious metallic elements such as Pb, Cd, As, Cr, Cu, Ni and Zn. The analyses data indicate that liquid fertilizer is free from aluminium and noxious metallic elements while ratio of nitrogen, phosphorous and potassium are close to the Indian NPK value.

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1. Introduction

Composite propellant is the most important class of solid rocket propellants being used in space as well as missile programmes [1]. It basically contains ammonium perchlorate (65–70%) as an oxidizer, aluminium powder (10–15%) as a metallic fuel and hydroxyl terminated polybutadiene (10–15%) as a pre-polymer binder along with certain process aids and diisocyanates based curatives. Large quantity of composite propellants is produced as waste due to expiry of missiles or rejection of propellant lots during manufacturing. The accumulation of composite propellants causes fire hazards, therefore, demilitarization and disposal of surplus and rejected propellants, explosives and energetic materials has been a requirements over the years. The most commonly methods of disposal are open burning and open destruction (OB/OD) of hazardous munitions and ingredients. Moreover, large scale reclamation of specific ingredients from propellants and warheads has been pursued only

when the manufacturing cost of strategic importance of ingredients are sufficiently high to justify the added recovery expenses.

Due to recent Environmental Protection Agency (EPA) restrictions on open burning of hazardous waste and munitions, NASA as well as other military organizations have adapted new regulations to protect environment from conventional means of destruction of propellants such as launch to destroy, static firing or open burning. The most widely used alternative to current OB/OD methods involves the use of incineration [2], molten salt oxidation (MSO) [3], supercritical waste oxidation [4], hydrothermal oxidation (HTO), wet air oxidation (WAO), supercritical biodegradation and chemical treatment processes. However, HTO, WAO or biodegradation processes are either costly or slow and not fully get exploited at large scale operations, discourage the adaption for field application/bulk treatment [5].

In view of limitations of above mentioned methods, there is a need to develop a method for disposal of energetic materials and composite propellant compositions which is cost effective, environmental friendly and disposal products can be further used. Therefore, an exhaustive literature survey has been carried out to study the developed methods/techniques in this field. The most widely used method for conversion of single base propellant (based on nitrocellulose) is alkaline hydrolysis in which the end product has been used as liquid fertilizer as ACTOSOL [6], a well patented product.

In continuation to this work further, the disposal of energetic molecules by alkaline pressure hydrolysis of nitrocellulose

Abbreviations: CP, composite propellant; HTPB, hydroxyl terminated polybutadiene; AP, ammonium perchlorate; ICP-AES, inductively coupled plasma-atomic emission spectrometry; IC, ion chromatography; NC, nitrocellulose; NPK, nitrogen–phosphorous–potassium; WAO, wet air oxidation; MSO, molten salt oxidation; HTO, hydro-thermal oxidation; OB, open burning; OD, open detonation.

* Corresponding author.

E-mail address: drmehilal@yahoo.co.in (Mehilal).

has also been reported [7]. In the same way, a chemical process has been developed for NC based explosives and propellants using 5–20% NaOH solution in the temperature range of 323–373 K [8]. The disposal of waste compounds such as ammonium perchlorate, RDX, HMX, NG, TNT and CL-20 having labile groups have also been carried out into environmentally acceptable products by hydrothermal decomposition methods [9]. The method for extracting ammonium perchlorate from composite propellant using anhydrous liquid ammonia has also been reported [10]. In another study, aluminium has been recovered from aluminized composite propellant using sodium methoxide and cross esterified alcohol to an extent of satisfaction [11]. Further to this, composite propellant slurry containing water in detergent were subjected to compressed air in supersaturated stream under severe atmospheric pressure to convert the propellant constituents into CO₂, H₂O, N₂O and other products [12].

The literature survey further reveals that there is not a single method has been reported to convert composite propellants into liquid fertilizer which is simple, safe and environmental friendly. Moreover, based on the earlier work reported elsewhere [13], i.e., conversion of NC into liquid fertilizer by treating with KOH followed by neutralization with orthophosphoric acid to produce nitrate, phosphate and potassium which are essential for plant growth, a new idea was conceived, developed and employed successfully to convert waste composite propellants into liquid fertilizer by digesting waste composite propellant with dil. nitric acid, followed by treatment with KOH and orthophosphoric acid.

In the following section, we report the conversion of waste composite propellant into liquid fertilizer by digesting with dilute nitric acid, followed by neutralization with KOH and orthophosphoric acid along with estimation of nitrate, phosphate and potassium in it.

2. Experimental

2.1. Materials

Waste composite propellant used for this study was from the rejected lot manufactured at HEMRL, Pune. Nitric acid and potassium hydroxide, A.R. grade, were procured from Merck, India while orthophosphoric acid-L.R. grade, was procured from Qualigens, India and used as such. All the standard reference samples of ICP-AES & IC were procured from E-Merck.

2.2. Characterization

2.2.1. Determination of metallic content

Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES), ULTIMA-2000 HORIBA, France, was employed for the determination of metallic content present in liquid fertilizer such as K, Al, Pb, Cd, As, Cr, Cu, Ni and Zn. It is an elementary analytical technique based on the procedure of excited atoms in plasma. An emission spectrum results due to electronic transitions from higher energy level to lower ones. When an atom subjected to plasma it emits photons that are characteristics to individual metallic element and number of emitted photons is proportional to the number of atoms in the elements under consideration.

3. Sample preparation

The sample was prepared by taking 1 g of liquid fertilizer into a 1000 ml volumetric flask followed by addition of 36 ml of concentrated nitric acid (69–70%) into it. After this, deionised water was added up to the graduated mark which provided the concentration of sample up to 1000 ppm. From the prepared solution, 10 ml of

solution was again taken into 1000 ml measuring flask and made up to the graduated mark by addition of 36 ml concentrated nitric acid followed by deionised water to make 10 ppm solution.

The reference samples were prepared from 1000 ppm standard solution for each element and made up to 10 ppm as per the method given for sample preparation and blank solution containing 2.5% concentrated nitric acid in 1000 ml measuring flask on addition of deionised water up to the graduated mark was also prepared.

4. Procedure

At the outset, plasma was kept on using ultra pure argon gas which provided the plasma temperature in the range of 8000–10,000 K. After the plasma stabilization, blank sample was injected through nebulizer to spray chamber which cleans the instrument. After this, reference sample (10 ppm) of the individual metallic element was injected through nebulizer to spray chamber where the sample got converted into aerosol to particles, to molecules, atoms, excited atoms and finally photon emission which gave corresponding spectrum of the elements. After confirmation of the reference sample spectrum, the test sample was run following the same procedure and accordingly the percentage of metallic elements was calculated by comparing the area of emission spectral lines by programmed software. The same procedure was repeated for each and every metallic element under test using different reference sample.

4.1.1. Determination of ion content using ion chromatography

Ion-chromatography (IC) is a form of liquid chromatography, measures the concentration of ion species by separating them based on their interaction with resin. The sample solution passes through a pressurized chromatographic column where ions are absorbed by column constituents. As an extraction liquid, known as eluent, runs through the column, the absorbed ion begin separating from the column. The area under the retention time of different species determines the ionic concentration.

Ion chromatography (IC) DIONEX make, Model No.-500, USA, was used for the determination of ions present in the liquid fertilizer. In the present IC, two types of column, i.e., anionic separation column (AS-11) and cationic separation column (CS-12) were utilized. The eluents used for anions and cations are 20 mM NaOH and 20 mM H₂SO₄, respectively, with 2.5 µl capacity injector. The maximum concentration of reference sample was kept about 5 ppm while that of the sample concentration was kept 10 ppm and beyond this concentration erratic results were observed. During the determination of ions, initially, the reference sample was diluted to 5 ppm from 1000 ppm standard reference sample on addition of deionised water for each ion procured from trade as per method of ICP-AES. The prepared reference sample was injected through the column and corresponding retention time curve were recorded. After this, the test sample having concentration of 10 ppm was injected. The comparison of area of peaks of reference sample and test sample with the help of software was calculated which directly gave the concentration of ions in a test sample.

4.1.2. Phytotoxicity study

Phytotoxicity is the degree to which a chemical or compound is toxic to plant. It can range from slight burning or browning of leaves to death of plant and can be caused by pesticides, herbicides, and solvents or impurities.

The prepared liquid fertilizer was also studied for phytotoxicity on germination index (GI) by taking approximately 2000 g of dry soil in 4 earthen pots properly labelled. The first pot contained no liquid fertilizer whereas 2nd, 3rd and 4th contains 12.5 ml, 25 ml

Table 1
Optimization of nitric acid concentration on digestion of composite propellant (5 g).

	Acid conc.						
	100	80/20	75/25	65/35	50/50	35/65	25/75
Volume of acid (ml)	50	40	37.5	32.5	25	17.5	12.5
Reflux time (h)	1.5	2.0	2.5	3.0	4.0	10.0	20.0
Appearance	Clear solution	Clear solution	Clear solution	Clear solution	Clear solution	Clear solution	Clear solution

and 50 ml liquid fertilizer, respectively. To each pot, 20 numbers of seeds were sown, regularly watered and kept in open area to observe the phytotoxicity effect.

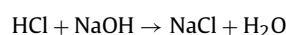
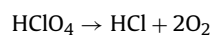
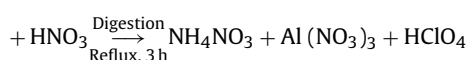
4.2. Procedure

10 g of waste composite propellant, already cut into small pieces, was transferred into a round bottom flask (cap-250 ml) equipped with a side condenser to another flask containing dil NaOH solution to absorb HCl coming out during digestion, followed by 60 ml of nitric acid (50 part conc. nitric acid + 50 part distilled water) and heated under reflux for 3 h for complete digestion of composite propellant. The clear and homogenous solution obtained by this process was cooled at room temperature and neutralized with drop by drop addition of 30 ml of 5 M KOH solution under stirring. During neutralization process, the white precipitate of $\text{Al}(\text{OH})_3$ started to appear. The precipitate thus obtained was filtered off and dried. The weight of dried product was 5.15 g (99% recovering of aluminium). Further to this, the alkaline solution obtained due to excess addition of KOH solution was again neutralized with orthophosphoric acid up to pH 7.2. The solution, thus, obtained was estimated for the presence of NO_3^- , PO_4^{3-} , NH_4^+ , Cl^- , K, Al, etc. and also used for phytotoxicity study.

5. Results and discussion

Initially, the small pieces of waste composite propellant was treated with concentrated HNO_3 (69–70%) subjected to get digested at room temperature and it was found that after keeping over a long period (4–5 days) only a partial digestion could take place. Therefore, to get a clear and homogenous solution, using concentrated nitric acid, it was kept under reflux. Due to use of concentrated nitric acid, it requires large volume of KOH for neutralization. In view of this, a systematic study was undertaken to digest waste composite propellant into homogenous solution using different concentration of nitric acid and accordingly different ratio of nitric acid using distilled water such as 80:20, 75:25, 65:35, 50:50, 35:65, and 25:75 were prepared and used. The different ratios of nitric acid, thus, prepared are shown in Table 1 along with their reflux time and volume of acids used. It is clear from the table that higher the concentration of nitric acid, higher the quantity of KOH required for its neutralization. In the same way lower concentration of nitric acid, i.e., 40 or 25 parts of nitric acid requires longer period for digestion even some times more than 20 h using 25 part nitric acid in the same ratio. Based on the different experiments, 50/50 ratio of nitric acid/distilled water was found suitable for complete digestion of propellant pieces in 3 h, hence 50/50 ratio of nitric acid/water was chosen for further study. Due precautions were also taken to trap HCl gas coming out during digestion after decomposition of HClO_4 by passing it into dil. NaOH solution which absorbs it completely. The overall digestion reaction can be expressed as

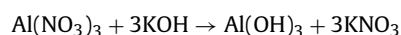
Composite propellant ($\text{NH}_4\text{ClO}_4 + \text{Al} + \text{HTPB} + \text{TDI}$)



Furthermore, HTPB used as prepolymer binder cured with toluene diisocyanate (TDI) to have required mechanical properties. During curing, hydroxyl groups of HTPB react with $-\text{NCO}$ groups of TDI to form urethane linkages, i.e., $-\text{NHC}(=\text{O})-\text{O}-$. The polyurethane linkage decomposes into smaller molecules during the digestion with nitric acid and a clear solution was obtained. It was also confirmed by taking FT-IR of the liquid fertilizer where it does not show any peaks of HTPB except nitrate groups and a broad peak of water. It was also confirmed by taking HTPB prepolymer only in 50% HNO_3 and heated under reflux for 3 h which resulted in a light yellow colour of precipitate and no clear solution was obtained, further confirms that it is polyurethane that breaks into smaller molecules and remains soluble in water as liquid fertilizer not HTPB, hence, degradation of HTPB does not arise.

5.1. Removal of aluminium nitrate from the digested solution

It is well known that aluminium is considered as detrimental for the growth of plants. Therefore, removal of aluminium from the digested solution was essential. Although sodium methoxide and cross esterified alcohol [11] has been used for the removal of aluminium from the composite propellant but this technique is not suitable here, hence different method and were explored to remove aluminium, i.e., either potassium hydroxide or ammonium hydroxide. Initially, NH_4OH (28–30%) was tried to neutralize digested product, however, the neutralization reaction is exothermic in nature. Therefore, dil. NH_4OH was tried, as neutralization with dil. NH_4OH is not very exothermic but in this case precipitation of Al nitrate as $\text{Al}(\text{OH})_3$ is incomplete. To cope up with this problem, potassium hydroxide was chosen to precipitate out aluminium nitrate as $\text{Al}(\text{OH})_3$. During this process, 30 ml of 5 M KOH solution was found sufficient to precipitate out complete aluminium nitrate from the digested mixture in the form of aluminium hydroxide at pH 6–8 and beyond this there is no precipitation of aluminium hydroxide occurs. The precipitate, thus, obtained was further filtered and dried. The precipitate contains 34.5% aluminium confirmed by ICP-AES which further confirms that the precipitate is in the form of hydroxide only. The overall reaction (theoretical percentage of aluminium in $\text{Al}(\text{OH})_3$ is around 34.6%) can be expressed by the following equation.



5.2. Neutralization of alkaline solution with orthophosphoric acid

Keeping in mind the requirement of phosphoric fertilizer for the flowering and fruits of plants, the excess of alkali present in the liquid was further neutralized with orthophosphoric acid (3–5 ml depending on the concentration of alkali present in the digested solution) and the neutralization reaction can be expressed as

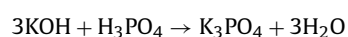


Table 2
Data on analysis of different ions present in the neutralized solutions.

Sr. no.	Ions	Percentage concentration of HNO ₃		
		100	50	25
1	NO ₃ ⁻	10–11	8–9	5–6
2	PO ₄ ³⁻	7–8	4–5	6–7
3	ClO ₄ ⁻	Nil	Nil	Nil
4	Cl ⁻	0.01–0.03	0.01–0.02	0.02–0.03
5	NH ₄ ⁺	5.0–7.0	3.0–4.0	3–4

5.3. Estimation of ions and metals in neutralized solution

The neutralized solution obtained after the treatment with orthophosphoric acid was used for the estimation of ions such as nitrate, phosphate, ammonium and chloride using ion chromatography and the results obtained are presented in Table 2. It is clear from the table that using 100% nitric acid, the percentage of NO₃⁻ and PO₄³⁻ are in the range of 10–11% and 7–8%, respectively, further support that essential macro ingredients required for the plants growth are present in digested solution. The other ions such as ClO₄⁻ is totally absent while Cl⁻ is present in trace amount. Moreover, the % content of NO₃⁻ and PO₄³⁻ for 50% and 25% HNO₃ acid are practically less than those for 100% nitric acid. The less value for NO₃⁻ and PO₄³⁻ is due to the use of low concentration of nitric acid.

Further to this, the estimation of aluminium as well as other metallic elements such as potassium K, Pb, Cd, As, Cr, Cu, Ni, and Zn were also estimated using ICP-AES and results obtained are presented in Table 3. The table clearly reveals the absence of aluminium content in digested solution, further confirms that 5 M KOH solution is sufficient to precipitate out aluminium as aluminium hydroxide completely from the digested solution which is detrimental to plants. It is also clear from the table that treated solution does not contain noxious metallic elements even in traces such as Pb, Cd, As, Cr and Cu whereas Ni and Zn are found in trace quantity of 0.0005%. The trace quantity of Ni and Zn is due to presence of impurities in aluminium powder used in the processing of composite propellant. Further to this, the percentage of potassium does not vary with concentration of nitric acid used for digestion.

5.4. Optimization of NPK content as per Indian standard

The neutralized solution after treatment with KOH and H₃PO₄ was further analyzed for NO₃⁻ as nitrogen, P as P₂O₅ and K as K₂O using different concentration of nitric acid and results obtained are presented in Table 4. The table clearly reveals that higher the percentage of nitric acid higher the percentage of N and P while potassium does not vary from higher concentration of nitric acid to lower concentration of nitric acid. It is well known that Indian standard of nitrogen, phosphorus and potassium (NPK) is in the

Table 3
Data on estimation of metallic elements in neutralized solutions.

Sr. no.	Metal%	Percentage concentration of HNO ₃		
		100	50	25
1	K	0.5–1.0	1.0–1.5	1–1.5
2	Al	Nil	Nil	Nil
3	Pb	Nil	Nil	Nil
4	Cd	Nil	Nil	Nil
5	As	Nil	Nil	Nil
6	Cr	Nil	Nil	Nil
7	Cu	Nil	Nil	Nil
8	Ni	0.005	0.005	0.005
9	Zn	0.005	0.005	0.005

Table 4
Data on analysis of NPK ratio for different concentration of nitric acid.

Concentration of nitric acid	N (%)	P as P ₂ O ₅ (%)	K as K ₂ O (%)	NPK ratio (%)	NPK ratio
100	7.56	5.68	0.783	54:41:5	10:7:1
80/20	6.85	4.696	0.988	55:37:8	7:5:1
75/25	5.94	2.31	1.32	62:24:14	5:2:1
50/50	4.60	2.61	1.49	53:30:17	3:2:1
35/65	4.48	2.89	1.30	52:33:15	4:2:1
25/75	4.09	4.686	1.30	41:47:13	3:4:1

ratio of 120:60:40, therefore, the percentage of nitric acid was optimized to get essential percentage of N, P and K with clean digestion. Accordingly, 50% concentrated nitric acid was found most suitable to get cleared digestion within 3–4 h and essential macro-nutrients obtained are very close to Indian standard of NPK, i.e., 120:60:45, respectively.

5.5. Phytotoxicity of liquid fertilizer

The prepared liquid fertilizer was studied for phytotoxicity on seed germination index and results obtained after sowing of seeds is presented in Fig. 1. It is clear from the figure that within the week, the germination of seeds started. The physical appearances clearly indicate that pot with 50 ml of liquid fertilizer initially shows higher shoot length and broaden green leaves in comparison to others. However, after a month, the growth of plants were not good and leaves also started becoming yellowish in colour which indicates that 50% concentration of liquid fertilizer is not suitable to plant growth.

Further to this, germination index (GI) of the seeds were also studied by sowing 20 seeds in each pot as per reported method elsewhere [14] by taking 25.0 ml, 12.5 ml and without liquid fertilizer in earthen pots. During the experiment, germination was recorded after 5 days and later on at 24 h interval and effect of fertilizer concentration on germination index was calculated as per following equation:

$$\text{Germination index (GI)} = \Sigma \left(\frac{Gt}{Tt} \right)$$

where Gt is the number of seeds germinated on day t and Tt is the number of days.

The data indicate that pot containing 25 ml of liquid fertilizer shows GI value 7.6 whereas pots containing 12.5 ml liquid fertilizer and without liquid fertilizer show GI value 5.6 and 3.9, respectively, clearly reveal that 25 ml of liquid fertilizer has better GI value and effect of phytotoxicity is not observed.

5.6. Economic analysis

Economic analysis of developed method was also carried out based on the chemicals used during the preparation of liquid fertilizer at 1 kg batch level without taking any extra cost. The overall estimated costs for 1 kg waste propellant are as follows:

Waste composite propellant (1 kg)	Chemicals/reagents required
Conc. HNO ₃ (50%)	3000 ml ~Rs. 600
Quantity of KOH for 5 M KOH solution	672 g ~Rs. 350
Orthophosphoric acid	450 ml ~Rs. 300
The total cost of chemicals used	~Rs. 1250 (\$25 only)

The present cost is based on lab scale taking all the ingredient of GR grade. However, this cost may come down drastically at large scale by procuring chemicals at bulk quantity level.

The prime interest to convert waste composite propellants into liquid fertilizer is to avoid open burning as it contaminate environment and use the liquid product as fertilizer, as it contains nitrogen, phosphorous, and potassium in the form of nitrate, phosphate and potassium superoxide which are essential macro-nutrients for the



Fig. 1. Study on phytotoxicity effect on seed germination Pot No. 1 contains 12.5 ml of liquid fertilizer. Pot No. 2 contains 25.0 ml of liquid fertilizer. Pot No. 3 contains 50.0 ml of liquid fertilizer. Pot No. 4 contains no liquid fertilizer.

growth of plants. Thus, for the development of root growth, potash fertilizer is required and in the growing stage, leaves and foliage require nitrogenous fertilizer while in the flowering to fruit stage, plants require fruits and flowering fertilizer, i.e., phosphate fertilizer. Further to this, our continuous efforts got succeeded to meet the Indian standard of NPK ratio using 50% nitric acid and the values are very close to it.

6. Conclusion

A successful method has been developed to convert waste composite propellant into liquid fertilizer by digestion with nitric acid followed by neutralization with potassium hydroxide and orthophosphoric acid. The developed liquid fertilizer contains NPK in the ratio of 126:60:45 confirmed by IC and ICP-AES, which is very close to Indian standard for NPK fertilizer being used. The developed liquid fertilizer is found non phytotoxic at lower concentration with high germination index. The developed method is simple, safe and environmental friendly and this technique may further be useful for the disposal of composite propellants on large scale level if developed suitably and waste stock piles of composite propellants may be exhausted one day for useful purposes.

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