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CHELTONOV M. M.[1], KYRYCHENKO O. L.[2]
[1]Corresponding author,
[2]State Enterprise Research-Industrial Complex «Pavlograd Chemical Plant»

EXPERIMENTAL STUDIES ON PROCESSING OF POLYBUTADIENE-BASED SOLID PROPELLANT TO EXTRACT AMMONIUM PERCHLORATE

Purpose. *The purpose of this work is to establish regularities of the leaching process and to determine parameters for leaching ammonium perchlorate from polymer crumb as solid propellant disposal products.*

Methodology. *The process of leaching ammonium perchlorate from the polymer matrix was carried out in a flask equipped with an agitator in a polymer matrix: water ratio of 1: 2, with such changing parameters as: temperature (20-80 °C), time of extraction process (1-4 h). Then, the obtained refined polymer matrix was filtered off, dried at room temperature, and weighed. The content of the chemical composition of the polymer matrix was determined in refined polymer matrix. The obtained polymer matrix was dried at temperatures of 70 ºC, 90 ºC, 100 ºC to constant weight, with intermittent weighing of dried samples.*

Findings. *It was determined that the degree of leaching of ammonium perchlorate from polymer matrix when the leaching time is 2-4 hours is maximum, both during the process at a temperature of 20 °C and at a temperature of 60 °C and 80 °C, respectively. It was found that the maximum degree of ammonium perchlorate leaching from polymer matrix was 82.3% in the experiment with the following modes - the process temperature was 80 °C, the speed of the mechanical stirrer was 400 rpm, and the leaching process took 4 hours. It was found that the most preferable temperature range for the drying of refined polymer matrix is 90 to 100 °C. It was determined that the refined polymer matrix is a substance which is not particularly dangerous for transportation and can be recommended for use as a filler in compositions of emulsion explosives, or for extraction of nitramine.*

Originality. *Establishing the regularities of ammonium perchlorate extraction from polybutadiene-based solid propellant, obtained from expired loaded motor cases.*

Practical value. *The data obtained after a detailed technical and economic analysis can be considered as the basis for the creation of a pilot industrial facility for the extraction of a water-insoluble component of solid propellant, i.e. ammonium perchlorate.*

Key words: *extraction, drying, ammonium perchlorate, polymer matrix, solid propellant.*

Introduction. In the process of utilization of solid propellant with an expired storage period using the hydro mechanical method [1-3], polymer crumb (polymer matrix) is formed. Solid propellant (SP) is a polyethylene-based polymer binder, filled with ammonium perchlorate, nitramine (1,3,5,7-tetranitro-1,3,5,7-tetraazocyclooctane), aluminum and process additives.

The working medium in the process of hydro mechanical utilization of solid propellant is water, which leaches up to 50% of ammonium perchlorate (AP) during extraction and shredding of solid propellant. Subsequent leaching of ammonium perchlorate, which is a valuable inorganic oxidizing agent and the production of which is unavailable in Ukraine, will increase its degree of extraction to 80% by weight.

Formulation of the problem. The purpose of this work is to establish regularities of leaching of ammonium perchlorate (AP) from polymer matrix obtained in the course of SP disposal. In order to assess the safe handling of the obtained refined PM after the leaching of AP, the parameters of its sensitivity to mechanical effects (impact, friction) were also determined.

Experimental. For studies there were used samples of shredding PM of solid propellant with a particle size shown in Table 1.

Table 1

The particle size of the initial solid propellant PM before leaching of ammonium perchlorate

Particle size	Fraction content, %
To 7x4x2 mm	31,7
From 8x4x2 to 10x4x2 mm	37,8
From 11x4x2 mm to 15x4x2 mm	28,9
More 15x4x2 mm	1,1

Tabl. 2 presents the chemical composition of PM before leaching ammonium perchlorate.

Table 2

The chemical composition of PM before leaching ammonium perchlorate

Component	Content of component before washing with water, %
Polymer binder	9,6
Ammonium perchlorate	28,1
Nitramine	29,5
Aluminum	25,9
Moisture	6,9

The process of leaching ammonium perchlorate from the polymer matrix was carried out in flask equipped with an agitator in a PM : water ratio of 1 : 2, with such changing parameters as: temperature (20-80 °C), time of extraction process (1-4 h). Then, the obtained refined PM polymer matrix was filtered off, dried at room temperature, and weighed. The content of the chemical composition of the polymer matrix was determined in refined polymer matrix. The obtained PM was dried in a Binder Fed 115 drying oven at temperatures of 70 ºC, 90 ºC, 100 ºC to constant weight, with intermittent weighing of dried samples.

In order to evaluate safety parameters in handling the derived product (refined PM), its sensitivity to mechanical effects (impact, friction) was determined. Impact and friction sensitivities were measured using a BAM Hammer 782-0000 and a BAM Friction Apparatus 781-0000 according to the methods [4].

Results and discussion. Fig. 1 and 2 show the polynomial dependencies of the degree of AP leaching from the polymer matrix on temperature and time, respectively. In accordance with the results obtained, the degree of leaching of ammonium perchlorate from PM in the temperature range of 80-100 °C reaches 61.5-76.3%, respectively. It was determined that the degree of leaching of ammonium perchlorate from PM when the leaching time is 2-4 hours is maximum, both during the process at a temperature of 20 °C and at a temperature of 60 °C and 80 °C, respectively.

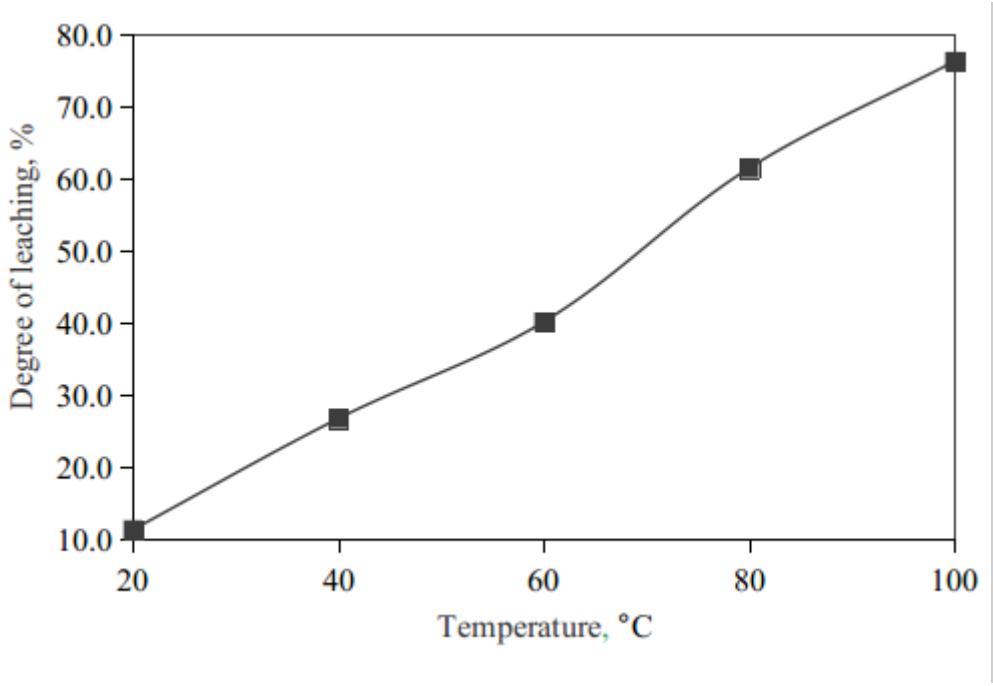

Fig. 1. **The dependence of the degree of AP leaching on temperature of leaching from polymer matrix (rotation speed of the mechanical stirrer of 400 rpm, time of extraction process of 1 h)**

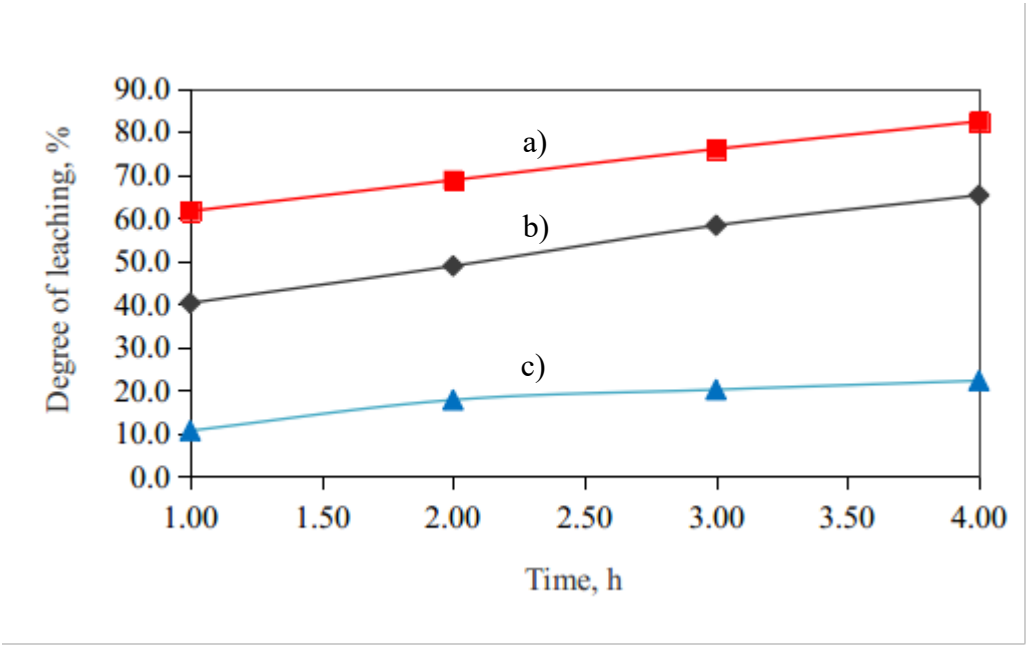

Fig. 2. **The dependence of the degree of leaching of AP on the time of leaching from PM: a) 80 °C, b) 60 °C, c) 20 °C (rotation speed of the mechanical stirrer of 400 rpm)**

It was found that the highest degree of leaching of 82.3% in terms of the target product during leaching from PM was achieved in the experiment which lasted for 4 hours, at a temperature of 80 °C, and a rotation speed of the mechanical stirrer of 400 rpm.

Under the specified conditions the optimum parameters for AP leaching from PM were determined as follows: the temperature is 60-80 °C, the extraction time is 2-4 hours.

Fig. 3 and 4 show the dependencies of PM drying after the leaching of ammonium perchlorate, and specifically the moisture content W of the polymer matrix versus time with a

change in the PM drying temperature, and drying rate versus moisture content of the polymer matrix at various temperatures, respectively.

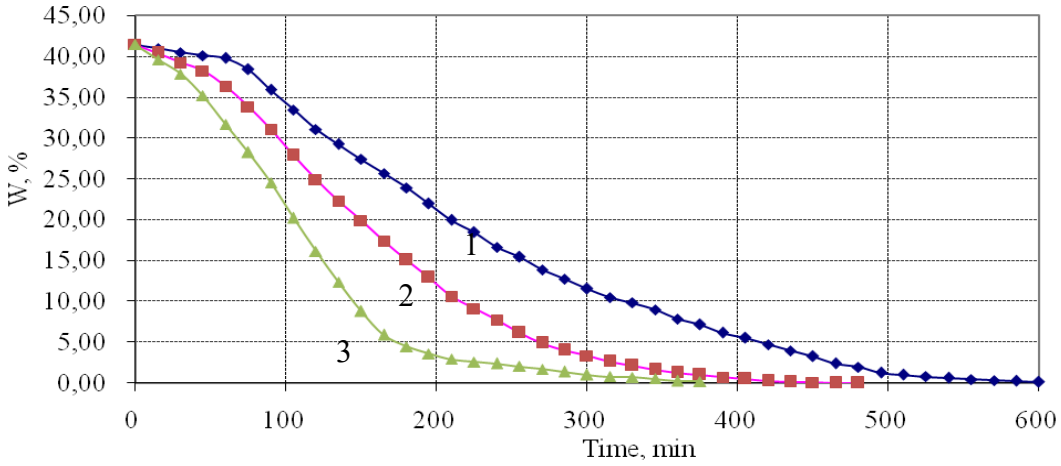

Fig. 3. **The dependence of the polymer matrix moisture content W on time with a change in PM drying temperature: (1) 70 ºC, (2) 90 ºC, (3) 100 ºC**

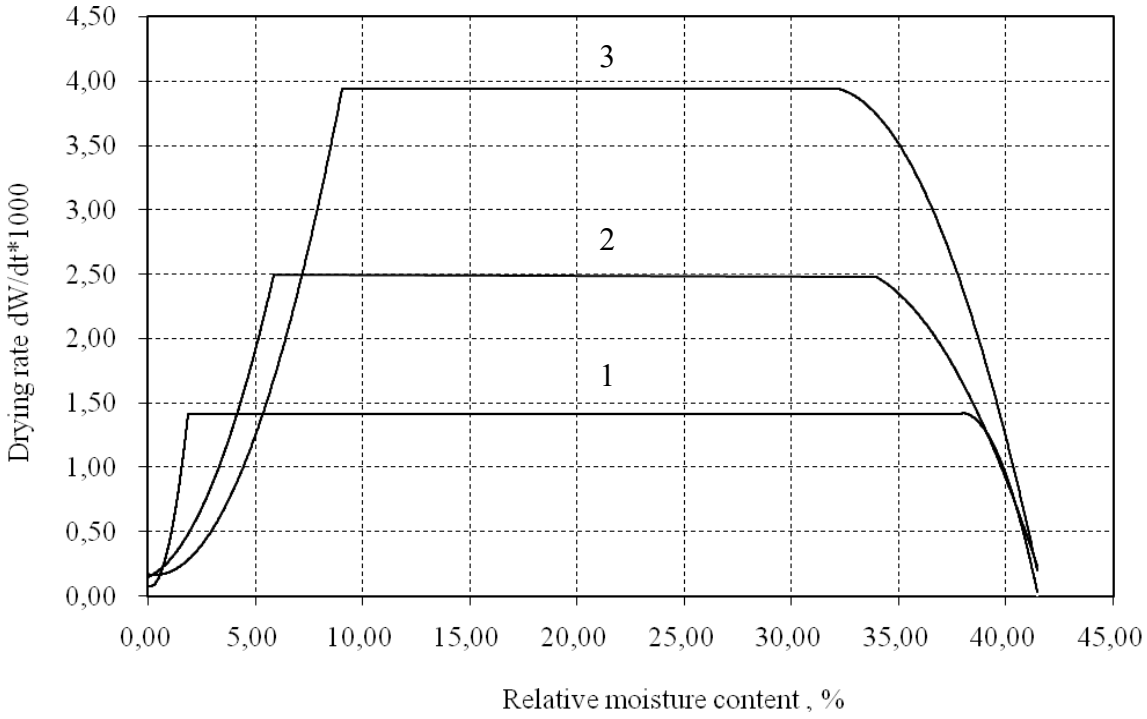

Fig. 4. **The dependence of drying rate on polymer matrix moisture content at various temperatures: (1) 70 ºC, (2) 90 ºC, (3) 100 ºC**

Thus, as can be seen from Fig. 3, the PM drying goes through two phases i.e. constant and decreasing drying rates, which are described in Fig. 3 with a straight line and an exponential function, respectively. The fastest PM drying to the constant weight takes place at a temperature of 100 ºC taking ~450 min. It is not advisable to dry the polymer matrix at temperatures of over 100 ºC, since at temperatures of above 115 ºC the nitramine, which is part of polymer matrix, can generate crystalline forms that are sensitive to mechanical impacts [9-12].

According to Fig. 4, with increase of the drying temperature from 70 ºC to 90 ºC, the drying rate rises 1.75 times, and with increase of the drying temperature from 90 ºC to 100 ºC, the drying rate rises further 1.6 times. Therefore, the most preferable temperature range for drying refined PM is 90 to 100 °C.

To assess the safety parameters in the handling of refined PM after leaching of the AP, its sensitivity to mechanical effects - impact, friction depending on moisture content was determined. Tabl. 3 presents the sensitivity results of refined PM depending on moisture content.

Table 3

Sensitivity results of refined PM depending on moisture content

Parameter name	Content of moisture in refined PM			
	41,45%	23,5%	16,9%	3%
Sensitivity to impact, J	>50	44,0	31,5	22.5
Sensitivity to friction, N	>360	>360	>360	>360

It was found that a refined polymer matrix with a moisture content of 3-41.45% is insensitive to friction (> 360 N), a decrease in humidity from 41.45 to 3% increases the sensitivity to impact to 22.5 J.

Refined PM's characteristics are below the minimum acceptable UN sensitivity requirements as for sensitivity to mechanical effects (≥2 J and ≥80 N), which proves that refined PM is a substance which is not particularly dangerous for transportation [4] and can be recommended for use as a filler in compositions of emulsion explosives [2], or for extraction of nitramine [5-8].

Conclusions. As a result of the studies, the dependencies of the influence of temperature and time on the degree of leaching of ammonium perchlorate from the polymer matrix of solid propellant are obtained. It was found that the maximum degree of AP leaching from PM was 48.3% in the experiment with the following modes - the process temperature was 80 °C, the speed of the mechanical stirrer was 400 rpm, and the leaching process took 4 hours.

It was found that the most preferable temperature range for the drying of refined PM is 90 to 100 °C.

Refined PM's (with a moisture content of up to 3%) characteristics are below the minimum acceptable UN sensitivity requirements as for sensitivity to mechanical effects (impact, friction), which proves that refined PM is a substance which is not particularly dangerous for transportation and can be recommended for use as a filler in compositions of emulsion explosives, or for extraction of nitramine.

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CHELTONOV MAXYM
ORCID: http://orcid.org/0000-0002-8077-1985
Head of Group for advanced technologies of energy materials and items,
State Enterprise Research-Industrial Complex «Pavlograd Chemical Plant»
dizel2008@meta.ua

KYRYCHENKO OLEKSIY
ORCID: http://orcid.org/0000-0002-1331-9323
Chief Technologist,
State Enterprise Research-Industrial Complex «Pavlograd Chemical Plant»
nelonme@gmail.com

ЕКСПЕРИМЕНТАЛЬНІ ДОСЛІДЖЕННЯ З ПЕРЕРОБКИ ТВЕРДОГО ПРОПЕЛАНТУ НА ОСНОВІ ПОЛІБУТАДІЄНУ З ВИЛУЧЕННЯМ ПЕРХЛОРАТУ АМОНІЮ

ЧЕЛТОНОВ М. М., КИРИЧЕНКО О. Л.
Державне підприємство «Науково-виробниче об'єднання «Павлоградський хімічний завод»

Мета. *Метою даної роботи є встановлення закономірностей і визначення параметрів вилучення перхлорату амонію з продуктів утилізації твердого ракетного палива.*

Методика. *Для вилучення перхлорату амонію зразки полімерної матриці обробляли водою при температурі 20-80 °С і перемішуванні 1-4 годин. Отриману рафіновану полімерну матрицю фільтрували, висушували при кімнатній температурі та зважували. Висушування проводили при температурах 70-100 °С. У рафінованій полімерній матриці визначали вміст перхлорату амонію, нітраміну, алюмінію, вологи, чутливість до механічного впливу (удару, тертя).*

Результати. *Відповідно з отриманими результатами ступінь вилучення перхлорату амонію з полімерної матриці в діапазоні температур 80-100 °С досягає 61,5-76,3 % відповідно. Виявлено, що ступінь вилучення перхлорату амонію з полімерної матриці максимальна, коли час вилуговування становить 2-4 год, як при проведенні процесу при температурі 20 °С, так і при температурі 60 і 80 °С відповідно. Встановлено, що максимальний ступінь вилучення перхлорату амонію з полімерної матриці твердого ракетного палива становить 82,3% в експерименті з наступними режимами - температура процесу 80 °С, швидкість обертання механічної мішалки 400 об/хв, час процесу екстракції 4 ч. Виявлено, що найбільш прийнятними показниками температури для сушіння рафінованої полімерної матриці є 90-100 °С. Визначено, що рафінована полімерна матриця не є небезпечною для транспортування і може бути рекомендована як домішка у складі емульсійних вибухових речовин.*

Наукова новизна. *Встановлено закономірності вилучення перхлорату амонію з твердого ракетного палива на основі полібутадієну, отриманого з двигунів з закінченим терміном експлуатації. Визначені параметри чутливості рафінованої полімерної матриці після вилучення перхлорату амонію до механічного впливу (удару, тертя).*

Практична значимість. *Отримані дані після детального техніко-економічного аналізу може бути розглянуті як основа для створення дослідно-промислового об'єкту вилучення водонерозчинного компоненту твердого ракетного палива - перхлорату амонію.*

Ключові слова: *екстракція, сушка, перхлорат амонію, полімерна матриця, тверде ракетне паливо.*

ЭКСПЕРИМЕНТАЛЬНЫЕ ИССЛЕДОВАНИЯ ПО ПЕРЕРАБОТКЕ ТВЁРДОГО ПРОПЕЛАНТА НА ОСНОВЕ ПОЛИБУТАДИЕНА С ИЗВЛЕЧЕНИЕМ ПЕРХЛОРАТА АММОНИЯ

ЧЕЛТОНОВ М. М., КИРИЧЕНКО А. Л.

Государственное предприятие «Научно-производственное объединение «Павлоградский химический завод»

Цель. *Целью данной работы является установление закономерностей и определения параметров извлечения перхлората аммония из продуктов утилизации твердого ракетного топлива.*

Методика. *Для извлечения перхлората аммония из полимерной матрицы, образцы полимерной матрицы обрабатывали водой при температуре 20-80 °С и перемешивании 1-4 ч. Полученную рафинированную полимерную матрицу отфильтровывали, высушивали, взвешивали. В рафинированной полимерной матрице определяли содержание химического состава - перхлората аммония, нитрамина, алюминия, влаги и чувствительность к механическому воздействию (удару, трению).*

Результаты. *Установлено, что максимальная степень извлечения перхлората аммония из полимерной матрицы составляла 82,3% в опыте со следующими режимами – температура процесса 80 °С, частота вращения механической мешалки 400 об/мин, время процесса экстракции 4 ч. Выявлено, что приемлемой температурой для сушки рафинированной полимерной матрицы является 90-100 °С.*

Научная новизна. *Установлены закономерности извлечения перхлората аммония из твёрдого ракетного топлива на основе полибутадиена, полученного из двигателей с истёкшим сроком эксплуатации. Определены параметры чувствительности рафинированной полимерной матрицы после извлечения перхлората аммония к механическому воздействию (удару, трению).*

Практическая значимость. *Полученные данные после детального технико-экономического анализа могут быть рассмотрены как основа для создания опытно-промышленного объекта извлечения водонерастворимого компонента твердого ракетного топлива - перхлората аммония.*

Ключевые слова: *экстракция, сушка, перхлорат аммония, полимерная матрица, твёрдое ракетное топливо.*