# PREPARATION NANO SIZED HMX BY USING ULTRASONIC WAVES

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### **ABSTRACT**

The researches done on particle size of explosive materials show that with reducing of particle size of explosive materials, reduced impact, friction and shock waves sensitivity. also by using of ultrafine explosive materials in propellant in comparison to larger particle size improved density, stability, calory and mechanical properties. Meanwhile the reduction of particle size of explosive materials influence on combustion mechanism of propellant and reduced burning rate and exponent pressure of different methods used for reduction of particle size, for example, milling, crystallization and etc. In this article for preparation of ultrafine HMX the method of solvent-anti solvent has been used by assistance of ultrasonic device. In optimum state the average size of particle is 106 nm and size distribution is narrow.

Key words: Sedimentary Crystallization, Ultrasonic, Reduce Particle Size, HMX

## INTRODUCTION

By the development of explosive materials with improvement performance and the reduction of sensitivity to mechanical stimuli such as impact and friction is one of the important researches goals in energetic fields. With reducing particle size of explosive materials decrease crystal defects and inclusion. This will reduce the sensitivity of explosive materials. Therefore safety of materials increases during handelling, processing, warehousing, and transportation. There searches done on the role of superfine particles of explosive materials in propellant show that with reducing of particles size increase density, calory, stability, and mechanical properties. Also one of the most important affecting issues on burning rate of propellant is the particles size of explosive materials. By using these ultrafine explosive materials in the propellant, the burning rate and exponent pressure decrease [1]. Various methods used for the reduction of the particles size of materials that the selection methods depend materials and expected properties such as the particles size distribution. Two general methods used for the reduction of the particles size up to down method

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including mechanical methods (grinding) and down to up method, including crystallization method (sediment crystallization fusion, supercritical fluid, and etc. In the reduction of particles size in mechanical technique reduction of particles size in mechanical technique, solids construction elements have been broken by mechanical forces and increasing the mass distribution happens. To reduce the particles size with mechanical method used various equipment such as pinned disk mill, jet mill, colloid mill, ball mill, etc., Mentioned methods have disadvantages such as static, electricity, pressure, and trituration that with considering the disadvantages, these methods are powerless and insecure to produce these ultrafine explosive materials [2-6]. Crystallization is the conversion of one or more material from liquid or gas state to crystalline state. This method used for modifying the physical properties of substance. In addition, the crystallization is a process for thickening pure material from solution, melt or gas phase one of the suitable methods for producing these ultrafine crystal of explosive materials is sedimentary crystallization method solvent-anti solvent. Because in method nucleation is high and high degree of saturation is seen. Although crystallization methods for preparing explosive materials have significant and advantages and crystals can grow slowly away from stress and obtain an ideal crystal. These methods have disadvantages such as [6-9]:

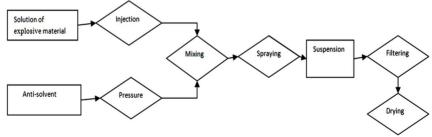
A: Sample should be soluble in one or more solvent solution and precipitate with reduction of temperature or increasing of anti-solvent.

B: Contaminate crystals to solvent and other solvate.

C: They spend too much energy.

D: Having lower yield (Because occurs in several stages)

A new method to produce ultrafine explosive is the kinetic spray crystallization that submicron size HMX and RDX are prepared with this method. The obtained particles size distribution with this method is very narrow. This schematic has been shown in *Fig.1* [10] Solvent/anti-solvent for spray solution by air into anti-solvent is used to produce ultrafine HMX The average of the obtained particles size with this method is 245 nm [1]. Also Mr. Lee and et al. by using sedimentary crystallization method have achieved HMX with an average particle size of 300 nm [11].



**Fig. 1** – Principle of kinetic spray crystallizing method [10].

In this research solvent/anti-solvent method has been used for producing ultrafine HMX by the assistance of ultrasonic device. In this method for controlling the crystallization process, the ultrasonic energy is used. To create ultrasonic waves, piezoelectric converters are used. These converters converted electrical energy into mechanical vibration with the same frequency. Mechanism to reduce the particles size of solid and emulsions by ultrasonic waves is cavitation. Cavitation is used as the center of nucleation of new crystals and growing. Also producing bubbles and bursting them caused trituration and grinding of solid particles [12].

## SAFETY

Generally by development of nanotechnology and producing nano particles in different scientific fields duet to the specific nature of nano particles produced and creating risk for human health, Considering the work, safety during producing, transportation, warehousing, and applying of nano particles should be considered. Therefore at the same time by growing the technology in producing nano particles, safety problems and diseases have been considered. In Considering the risks of toxicity of nano material in three contact areas in human body with the environment can be mentioned: skin, respiratory system, lungs, digestive system (mouth-esophagus). These three elements are the enteries for the nano particles in human body and from the way it can be entered in blood stream and internal systems and nervous. Of course it should be mentioned that the safety hints for producing nano explosive materials are not available. There fore, the respiratory mask, gloves, and suitable ventilation system can be away to control damages [13].

# **EXPERIMENT**

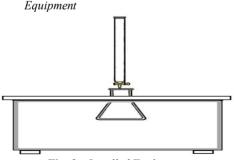


Fig. 2 – Installed Equipment

Ultrasonic device used H80 model is made in Elma Company of Germany. To determine the morphology and particle size of electronic Microscope XL30 manufactured in Philips Company of Dutch has been used. Installed equipment for doing the experiment is shown in *Fig. 2*.

Procedure

In this method certain concentrations of HMX in acetone solvent prepared at room temperature (*Table 1*) and then by using of a burette of 0.5 cc are injected into an erlenmeyer flask containing anti-solvent (water) that its

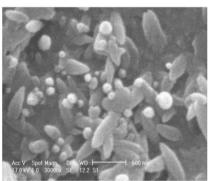
temperature is zero degrees Celsius. Solvent injection rate is about 0.7 ml per minute. Ultrasonic frequency is 80 Hz and its power is 750 Watt.

	Sample name	Amount of HMX (gr.)	Solvent (acetone) (ml)	Anti- solvent(water)(ml)	Anti-solvent Temperature (centigrade degree)
	Sample-1	0.1	5	80	0
	Sample-2	0.1	10	80	0
	Sample-3	0.1	20	80	0

Table 1 - Characteristic of produced samples

## RESULTS AND DISCUSSION

SEM image and the particles size distribution graph of sample respectively have been shown in Fig. 3 and Fig. 4. Also SEM image and the particles size distribution graph of sample-2 in Fig. 5, Fig. 6 and sample-3 in Fig. 7, Fig. 8 have been shown respectively.



**Fig. 3** – SEM image of sample -1

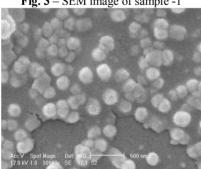
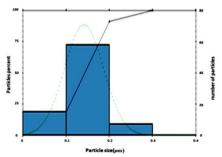


Fig. 5 - SEM image of sample -2



**Fig. 4** – The particles size distribution by analysis of sample -1

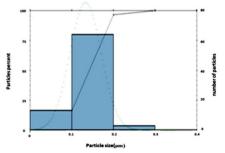
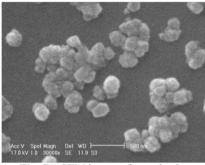
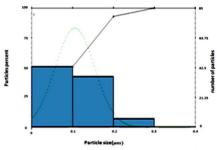


Fig. 6 – The particles size distribution by analysis of sample -2





**Fig. 7** – SEM image of sample -3

**Fig. 8** – The particles size distribution by analysis of sample -3

According to the images and the particles size distribution graph, it is observed with reduction of concentration samples can be achieved to smaller particle size. The best particle size is related to sample -3 that have the lowest concentration among the prepared solutions. The obtained average particles size is 106 nm. The reason for the reduction of the particles size with reduction of concentration is expressed with the degree of relative supersaturated theories. In other words, with reduction of samples concentration increases the degree of relative super saturation. Consequently, nucleation rate is more than nuclei growth and obtained smaller particle size. The results about the obtained particles size have been shown in *Table 2*.

**Table 2** – The analysis of particles size distribution

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	Sample name	Number of particles	Average of particles	Minimum of particles size	Maximum of particles size	
	Sample-1	80	0.0141	0.061	0.261	
ĺ	Sample-2	85	0.133	0.043	0.0210	
	Sample-3	85	0.106	0.037	0.0261	

### CONCLUSION

By the development of science in various fields application of nano materials has been expanded considerably. Among its applications can be mentioned to pharmaceutics, explosive materials, and etc. Various methods such as grinding, crystallization and sol-gel are used for producing materials in nano scale. One of the most common methods for producing nano-size explosive is sedimentary crystallization or solvent/anti-solvent method. Ultrasonic method combining this method and it can be a good idea for producing materials in nano scale. In this method different factors can influence on the particles size and the particles size distribution production. One of these factors is the concentration of produced solution. Investigation results showed that by reduction of solution concentration can achieve to smaller particle. In

optimum state, the average of particle size was 106 nm One of the most important factors for producing of the nano particles that should be considered is agglomeration of them that it has been a little reduced by using of ultrasonic. But research should be done to prevent agglomeration of nano-sized particles.

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