FLUIDIZED BED COATING OF AMMONIUM DINITRAMIDE (ADN)

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Abstract

Ammonium dinitramide (ADN) is a promising oxidizer, which could be applied e.g. in solid rocket propellants. The drawback of ADN is the less compatibility with the curing agents used in common binder systems. This incompatibility should be overcome in future by coating of ADN particles.

The aim of this work was to enhance a particle coating technology, which is suitable to process the hygroscopic and sensitive ADN-Prills, made by emulsion crystallization. In this context the fluidized bed technology, which proved to be useful for coating particulate explosives an oxidizers like ADN, has been established at ICT. The process parameters have been investigated and adjusted. Some different coating materials, e.g. polymers, were tested. The investigations of the coated particles by particle size analysis and microscopy have shown that the coating process is applied successfully to particulate energetic materials. It is to be seen that particles, with a mean particle size of 120 µm, can be coated separately and with uniformly thin layers.

1 Introduction

Ammonium dinitramide (ADN) is a high-performance, non-polluting oxidizer with the ability to replace ammonium nitrate or ammonium perchlorate in solid rocket propellants. Other possible applications for ADN are gas generators or underwater explosives.

The disadvantages of ADN are the insufficient thermal stability and particle morphology and the less compatibility with ordinary used polymer binder systems. Therefore the properties of the raw ADN from synthesis has to be improved concerning the particle size and shape by the ADN-prilling process [1] and concerning the stability by embedding stabilizers into the ADN-prills [2].

Nevertheless the incompatibility of ADN-prills and the isocyanates in common binder system persists. Therefore the aim of this work was to achieve a particle coating that is suitable to protect the ADN-prills sufficiently against the needed isocyanates. Besides of this the improvement of the compatibility, the other reasons for coating energetic materials are the protection against environments conditions (e.g. humidity and radiation), decreasing the materials tendency to agglomeration and the reduction of sensitivity.

Conducted studies concerning the process technology showed that the fluidized bed technology is suitable for coating particulate energetic materials like ADN.

2 Particle coating using the fluidized bed technology

A fluidized bed is formed when a quantity of a solid particulate substance, usually present in the process vessel, is forced to behave as a fluid. This is usually achieved by the introduction of pressurised gas that flows upward through the perforated bottom plate of the process vessel. The resulting phenomenon is called fluidization. Fluidized beds are used for several purposes, such as fluidized bed granulation, -drying or -particle coating [3].

In case of fluidized bed coating the particles that form the fluidized bed are the core material whereas the shell material is spayed onto the fluidized particles as a solution. This solution contains the solid shell material solved in aqueous or organic solvent. This solution, usually atomized by two flow nozzles, gets together with the moved core particles and spreads on its surface. Caused by the flow rate and the

temperature of the process gas the solvent evaporates whereas the solid coating material remains as a thin closed film on the surface of the core particles. It is very important to adjust the process parameters correctly, because a balance between the input of coating material together with the solvent and the evaporation rate of the process gas is necessary to get high quality coating results [4, 5]

3 Fluidized bed coating apparatus

Standard fluidized bed plants are often used for pharmaceutical or food processing applications. Associated with the research fields of the energetic materials department of ICT a modification of a standard laboratory apparatus was necessary. As it must be possible to work with dry particulate oxidizers and explosives and also with hygroscopic materials, the adapted fluidized bed coater (Figure 1) fulfils the following requirements:

- Remote control
- The process unit is positioned in a safety work room
- Explosion-proof electrical installations
- Resistance to organic solvents
- Suitable to hygroscopic materials

Fig. 1: Fluidized bed coating apparatus (Hüttlin, Mycrolab)

The process vessel wherein the fluidized bed is generated is seen in Figure 2. There are two different vessel sizes. A small one for 50 – 300 ml bulk volume and a bigger one for 250 – 1000 ml bulk volume.

Figure 2: Process vessel of the fluidized bed apparatus

Figure 3 shows the perforated bottom plate with the concentrically installed two flow nozzle that injects the coating fluid.

Figure 3: Perforated bottom plate

The fluidize bed coating machine is equipped with a computerized process control that affects:

- Temperature of the incoming process gas
- Flow rate of the process gas
- Flow rate of the dissolves coating material

Additionally measured parameters are:

- Temperature oft the fluidized bed
- Temperature of the outgoing gas
- Pressure difference at the filters
- Concentration of the evaporated organic solvents (compared to the lower explosion limit)

4 Experimental and results

Core materials for the experiments are ICT made products:

- ADN-Prills, produced by the emulsion crystallization process [1]. Mean particle size 121 µm.

- Spherical ammonium nitrate particles; SCAN (spray crystallized ammonium nitrate). Mean particle size 150 µm.

The pure SCAN without coating is show in Figure 4. In comparison to this coated particles are pictured in the Figures 5 and 6. It is to be seen that the particles are coated separately and uniformly.

Figure 4: Pure SCAN without coating

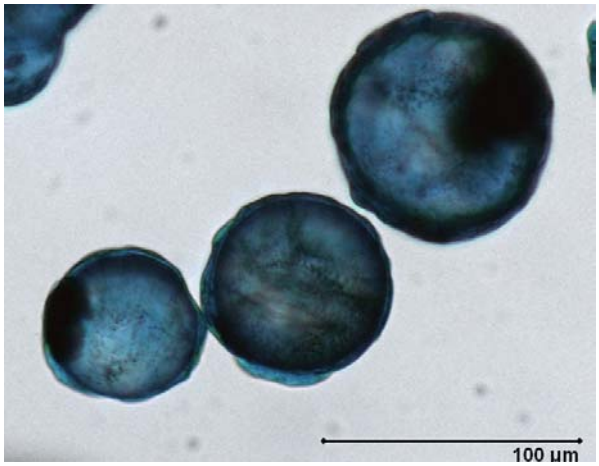

Figure 5: SCAN coated with a polymer (from organic solvent)

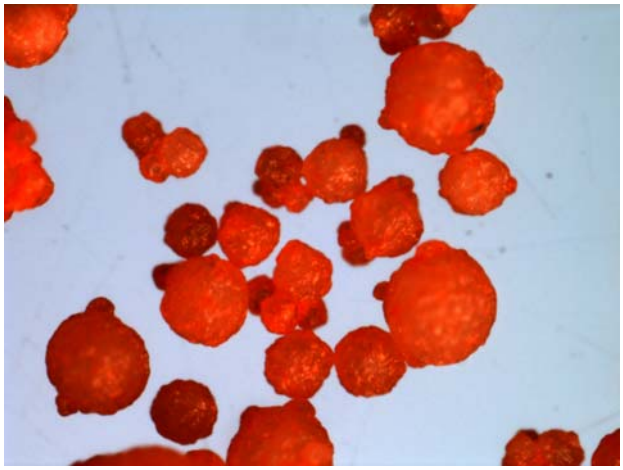

Figure 6: SCAN coated with a polymer (from aqueous system)

Figure 7 shows the ADN-Prills without coating whereas in figure 8 and 9 homogenous coated ADN-Prills can be seen.

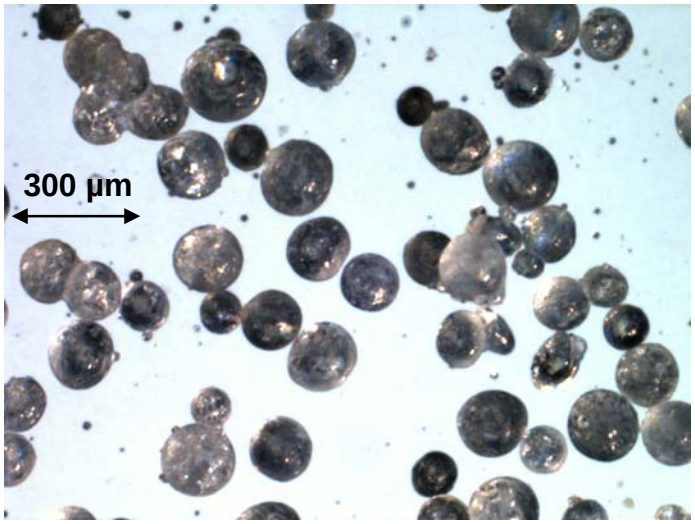

Figure 7: ADN-Prills without coating

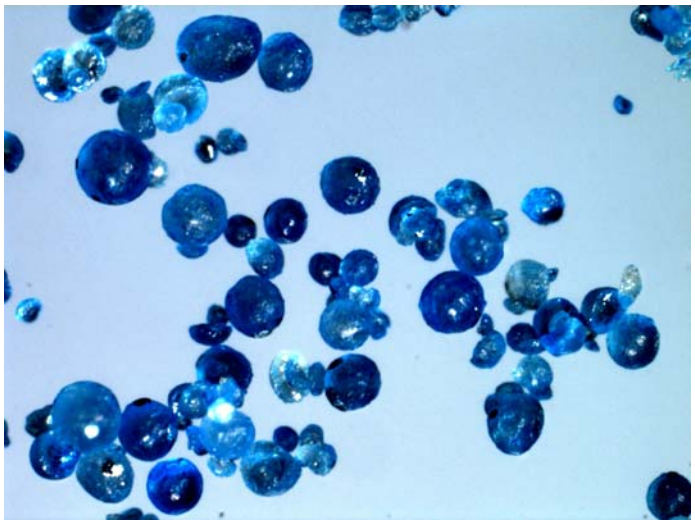
Figure 8: ADN-Prills coated with a polymer (from organic solvent)

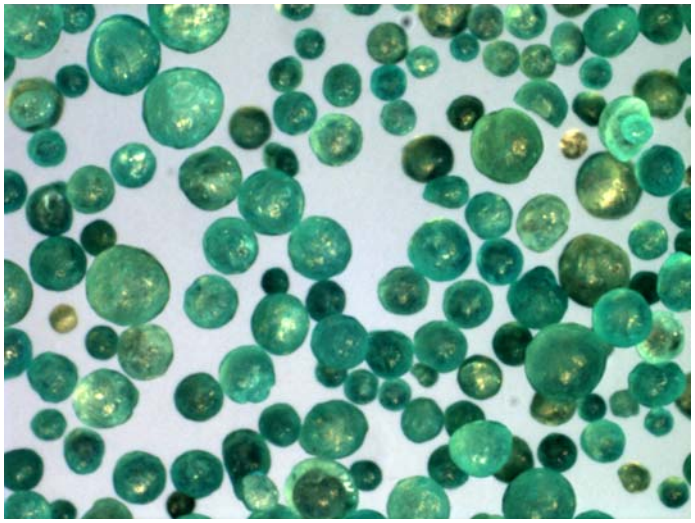
Figure 9: ADN-Prills coated with a crystalline substance (from organic solvent)

The coated ADN-Prills are furthermore investigated by particle size analysis. The mean particle size and the particle size distribution are measured by laser light diffraction (Malvern Mastersizer S). Regarding the results of the particle size analysis (table 1) relating to the microscope pictures (Fig 7 – 9) it is to be seen that the coating result are of good quality. According to VDI-Richtlinie 3491 the particle size distribution K is a dimensionless ration with the following meaning:

$K \leq 0{,}14$	monodisperse
$0{,}14 \leq K \leq 0{,}41$	quasi-monodisperse
$0{,}41 \leq K$	polydisperse

The fact that the mean particle size and the particle size distribution is nearly not influenced by the coating process shows that neither agglomeration nor particle breakage occur. Agglomeration would result in bigger mean particle sizes und a wider distribution, whereas particle breakage would lead to smaller mean particle sizes and also wider distribution. Both phenomena would also easily be seen in the microscope pictures.

Table 1: Particle size analysis of coated an uncoated ADN-Prills

	ADN-Prills without coating	ADN-Prills coated with a polymer	ADN-Prills coated with a crystalline substance
mean particle size [µm]	121	108	125
particle size distribution K [-]	0,36	0,29	0,24

5 Conclusions

The fluidized bed technology used for coating particulate energetic materials, especially ammonium dinitramide (ADN), has been established at ICT. The process parameters has been investigated and adjusted. Different coating materials were tested. The investigations of the coated particles by particle size analysis and microscopy have shown that the coating process is applied successfully to particulate energetic materials.

6 References

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