Research on Spray Granulation of Delay Explosive

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Abstract. In view of the problems existing in the delay explosive granulation, a spray granulation technique was proposed. By spray granulation with Wolfram type delay explosive, the influence of safety related factors such as temperature and moisture on granulation was studied. SEM was used to analyze the morphology of the samples. Results demonstrate that the particles of the delay composition were spheres with diameter of 100–400 µm. A hollow ball is formed due to the rapid evaporation of water. The sample has good dispersibility and the moisture content is 4.42%.

Keywords: Delay explosive; spray; granulation; technique.

1. Introduction

1.1. Introduction to Delay Explosive
Delay explosive is a time control element which ignites or detonates next elements in the sequence by controlling the time of action through stable combustion. Delay explosive has been widely used in weapon, aerospace, rocket missile, mining, and civilian blasting.[1]

Sieve granulation, as the most commonly used granulation method of delay explosive, mixes raw materials with a binder and dries the mixture to a humidity suitable for granulation, and obtains delay explosive with the required particle size through sieving. In the process of sieve granulation, delay explosive raw materials and excipients are not isolated from employees, which raises higher requirements for fire protection and explosion protection. Workers are exposed to toxic and harmful heavy metal raw materials and excipients, leading to higher risks of occupational diseases. Moreover, such delay explosive has the disadvantages of low mechanical strength poor dispersion, and is likely to generate fine powder during the use process [2-3].

1.2. Introduction to spray granulation
Spray granulation is a granulation method which uses a nebulizer to disperse the raw material liquid into mist droplets and obtain the products through hot air drying. Spray granulation is suitable for drying heat-sensitive substances, such as biological products, biological pesticides and enzymes preparations. Only when sprayed into mist particles are the materials exposed to high temperature and instantly heated to keep the active materials from deteriorating even after drying. Spray granulation has been extensively used in various fields such as ceramics and superconductors, biochemicals, dyes, soaps and detergents, food adhesives, oxidation Materials, bone meal, dentifrice, etc.
Spray drying indicates that the air enters the air distributor on the top of the dryer after being filtered and heated, and the hot air enters the drying chamber spirally and uniformly. The suspension is sprayed into misty liquid globules through the top of the tower, which are exposed to the hot air and dried to the finished products in a very short period of time. The finished products are continuously output from the bottom of the drying tower and the cyclone, and the exhaust gas is evacuated by the induced draft fan.[4-5]

1.3. Key Issues of Delay Explosive Spray Granulation
(1) Delay explosive is flammable and explosive, and has the risk of burning at high temperature. Therefore, temperature control is the key to product quality and safe production. (2) Delay explosive has higher requirements for product fluidity, which is mainly determined by particle size (or sphericity). The higher the sphericity, the better the fluidity. (3) Suspension of solid raw materials is the key to spray granulation.

2. Experimental Materials and Experimental equipment

2.1. Experimental materials
Raw materials for delay explosive: Wolfram type delay explosive manufactured by Weili Chemical Co., Ltd in Yibin, Sichuan was experimented. Industrial-grade water glass was used as a dispersant, and polyvinyl alcohol (PVA1788) was used as a binder.

2.2. Experimental equipment
HZ-1500 spray desiccant purchased from Shanghai Huizhan Experimental Equipment Co., Ltd., 8411 electric standard sieve shaker and German ZEISSEVO18 Zeiss scanning electron microscope were used in the experiment.

3. Experimental design

3.1. Mixing Process—Preparation of Suspension
A certain amount of tungsten powder, barium chromate, potassium perchlorate and additives were weighted according to the formula, and added to the beaker. Water was added in proportion, and dispersant and binder were added and stirred for several minutes to prepare the suspension.

3.2. Spray granulation
The spray granulation equipment was started. The nozzle was 1.0mm, the spray pressure was 0.15MPa, the dry air inlet temperature was 240 °, the feed volume was 15mL/min. The dried products enter the aggregate bottle through the cyclone separator.

3.3. Analysis and detection

3.3.1. Analysis of fluidity. The angle of repose was determined according to the standard of “GB 11986-1989 surface active agent—Powders and Granules—Measurement of Angle of Repose”. The smaller the angle of repose, the smaller the friction, and the better the fluidity. In general, the fluidity is good when $\theta \leq 30^\circ$, and the fluidity in the case of $\theta \leq 40^\circ$ can meet the requirements in the production process.

3.3.2. Granulation rate. The particle size of the delay explosive was measured according to “GB/T 1480-2012 Metallic Powder— Determination of Particle Size by Dry Sieving”. The percentage of 40-80-mesh delay explosive to the total particles was taken as the granulation rate.
4. Result discussion

4.1. Drying temperature control of delay explosive

According to the spray granulation drying process, if the outlet temperature is not higher than the limit temperature, overheat will not occur during the spray drying process. By measuring the temperature of the delay explosive and the hot air entering the granulation equipment in the spray granulation process, the time varying curves of the temperature of the delay explosive and the hot air were simulated, as shown in Figure 1.

Figure 1. Time-varying curve of the temperature of delay explosive and hot air

Figure 2. Relationship between solid content of slurry and water content of delay explosive
As can be seen from the hot air temperature change curve in Figure 1, the delay explosive slurry forms small droplets through atomization. The droplets quickly absorb the heat of the hot air, and its temperature rises to point B, and then enters the constant-speed drying stage BC after evaporation of latent heat. When the delay explosive is dried to the critical point C, its solid surface gradually forms and the surface temperature rises rapidly. The hot air temperature change curve shows that after the hot air enters the dryer, evaporation of the slurry absorbs heat. The air temperature drops rapidly until it is close to the temperature of the delay explosive, and the two curves intersect at point D. In this case, the delay explosive is discharged outside the dryer with the hot air, and the temperature at Point D is the outlet temperature.

Therefore, the temperature of the delay explosive can be controlled by controlling the outlet temperature. Factors such as slurry feed volume, solid content, hot air temperature and flow directly influence the outlet temperature of the delay explosive. If the outlet temperature is too low, the moisture content of the delay explosive is high, and the delay explosive particles will stick together, thus affecting the granulation rate.

4.2. Factors influencing the moisture content of delay explosive

The dispersant was added, and the insoluble delay explosive component was formed to dispersing slurry for granulation. The relationship between the solid content in the slurry and the water content of the delay explosive is shown in Figure 2.

The residual moisture content of the delay explosive directly affects production safety. There are clear requirements for moisture content of delay explosive has clear requirements. Study results demonstrate that increasing the solid content of slurry can decrease the moisture content of delay explosive due to the decrease of the evaporation load. In Figure 3, the solid content of the slurry increases, the moisture entering the granulation dryer decreases, and the moisture content of the delay explosive is reduced. When the solid content of the slurry is higher than 65%, the viscosity of the slurry rapidly increases, and the nozzles are likely to be blocked.

Meanwhile, the inlet temperature can reduce the moisture content in delay explosive. Figure 3 shows the relationship between different inlet temperatures and the moisture content of delay explosive.

![Figure 3. Relationship between inlet temperature and moisture content of delay explosive](image-url)
As shown in Figure 4, the temperature gradually increases, the moisture gradually decreases, and the temperature is higher than 260℃. In fact, since the droplets expand in spray granulation, the evaporation becomes extremely fast when the inlet temperature increases to a certain degree. The droplets will break up, disintegrate and disperse due to excessive expansion. Finally, undesired powdery substance will be generated. Figure 3 describes the relationship between the inlet temperature and the moisture content of the product.

4.3. Analysis of Particle Morphology

4.3.1. Analysis of Solid Ball Formation. The optimization condition of spray granulation is obtained through experimental analysis of the influencing factors: the solid content is 60%, the feeding amount is 20ml/min, the binder is water glass, the additive amount is 1.5%, the pressure is 120kPa, the 1.0mm nozzle is selected, the hot air temperature is 240 °C, and the spray granulation is carried out. The obtained delay explosive has an angle of repose of 27.5 °, a bulk density of 1.704, and good fluidity. The moisture content is 4.42% and the granulation rate is 48.9. By analyzing the product through SEM, the morphology of the particles is shown in Figure 4.

Figure 5 indicates that the delay explosive particle is spherical, and the particle size is mainly distributed in the range of 100-400 μm. Due to the high water content of the delay explosive, some small particles and large particles are bonded together.

After the atomized droplets enter the drying chamber, the moisture content decreases due to the high solid content of slurry, the drying air temperature is suitable, the water evaporation rate is well controlled, and a solid ball is formed. Of course, the so-called solid ball is relative. Spray granulation is theoretically hollow spheres, but the degree of hollowness varies.
4.3.2. Analysis of Formation of Hollow Spheres. In the case of high temperatures, small feed volumes, and low solid content of slurry, the delay explosive obtained by spray granulation has poor liquidity. The picture taken through a photomicrograph is shown in Figure 6.

As shown in Fig.6, broken spherical shells occur in spray granulation of delay explosive. The formation mechanism is that the evaporation rate of the droplet surface is greater than the solvent
diffusion rate at high drying temperature. When the water on the droplet surface evaporates, a gel shell is formed because the water in the center does not diffuse. As the evaporation continues, the internal solution migrates from the inside of the droplet to the droplet surface under the action of the capillary. After evaporation, the solute precipitates on the surface and fills the space between the grains, forming a smooth surface. As the drying progresses, the droplet surface is dried and solidified, the core inside the droplet shrinks, and the temperature of the particles rises and exceeds the boiling point of the solution. Evaporation occurs inside the particles. The porosity is too small to make the water vapor discharged. When the pressure in the shells rises to exceed the strength at local part, there will be vent holes, and damaged spherical shells are formed.

5. Conclusion

5.1. Conclusion
With Wolfram type delay explosive as raw material, delay explosive is configured as a suspension slurry for spray granulation, and the obtained product has an angle of repose of 27.5°, a bulk density of 1.704, and good dispersibility. The moisture content is 4.42%, and the granulation rate is 48.9%. The delay explosive is hollow sphere of 100-400 μm with a loose structure on the surface.

The residence time of delay explosive in granulation is short, and the maximum temperature is related to the solid content of the slurry, the feed amount and the hot air temperature.

5.2. Recommendation
There are several problems with spray granulation of delay explosive. In the spray granulation process, the delay explosive bonds to the spray cavity (drying chamber), and the temperature of the spray cavity surface is higher than that of the delay explosive. Moreover, the granulation rate is small, and the production cost is high.

In this experiment, a small spray drying equipment was used, which was designed based on food spray granulation. The spray equipment was designed and modified according to the physical properties of the delay explosive, which greatly enhanced the granulation rate of spray granulation, reduced or solved sticky walls, and developed a new field for delay explosive granulation.

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