Analysis of Sulfur Granulating Process

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I. Description

Owing to increasingly stringent environmental regulations and increasing demand for sulfur, there has been an upward trend in desulphurization and sulfur granulating.

At present, more than 40 million tons of sulfur has been degassed as byproduct of petroleum and natural gas. If sulfur from desulphurization by coal chemical industry, smelting industry and power generation industry is counted, annual throughput of sulfur will be more than 40 million tons. Furthermore, sulfur has much broader scope of application. However, due to special physical and chemical characteristics of sulfur, efforts must be made to transform a majority of liquid sulfur into sulfur granules so that sulfur can be transported, stored and used safely, reliably and environment-friendly. To sum up, finished product of sulfur granules is stable, dust-free, environment-friendly and excellent in flowability. Therefore, sulfur granulation is a must for relevant industries.

However, how should we select suitable sulfur granulating method and process for different characteristics of production, different process, different equipment scale and different product application fields in order to better satisfy different production requirements?

I’d like to describe sulfur granulating process by introducing my own technical practice for your reference and discussion.

II. An Introduction to Granulating Process

Sulfur is granulated for the following two purposes: firstly, to rapidly and effectively transform liquid sulfur into solid sulfur and satisfy the requirements of production process, safety and environmental protection; secondly, to obtain stable and uniformly sized sulfur in the process of transforming liquid sulfur into solid sulfur and make it easier to transport, store and use sulfur.

At present, four processes are used to granulate sulfur: rotary belt cooling granulating process, air cooling tower granulating process, water cooling granulating process and spray granulating process, which are introduced as follows:
1. Rotary Belt Cooling Granulating Process

Rotary belt cooling granulating process: to take advantage of low melting point of sulfur and evenly load liquid sulfur on to steel belt moving at a uniform speed using special distributor according to scope of viscosity of molten sulfur; compulsory cooling by continuous spray device on the underside of the steel belt enables sulfur to be quickly cooled, solidified and formed as they are being conveyed; please see Figure 1.

Main features:

1) Distributor rapidly and regularly divides continuous liquid sulfur into liquid drop; viscosity and surface tension of molten sulfur enables liquid drop to form hemispherical uniform granule. Particle size of sulfur granule is controlled through coordinate regulation of rotation speed of distributor and flow rate of sulfur. Yield rate of sulfur granule reaches nearly 100% without screening and returning sulfur; the process is simple and performance proven. Please see Figure 2 for working principle of distributor.

2) Thin steel belt cooler is sprayed on the underside with water carefully positioned to ensure uniform cooling, enabling liquid sulfur to be quickly cooled, solidified and formed as they are being conveyed; the heat released during cooling and solidification is eliminated through thin steel belt, avoiding contact of sulfur with cooling water and ensuring moisture content of sulfur granule.

3) As rotary steel belt changes its direction at discharge end, enabling junction surface of solidified sulfur granule and steel belt to be easily peeled, little dust occurs when sulfur is being discharged, and particle shape is ensured; this contributes to improvement of operating conditions and environment.

4) Automatic control of total process can be exercised according to technological requirements, ensuring that operating parameters (including temperature, flow rate and pressure) remain stable, and that sulfur remains in a stable state, and that system operation is stable and reliable.

5) Maximum productivity of a rotary belt cooling granulator only reaches 6,000 kg/h, which is quite low for a large-scale sulfur recovery device. Parallel operation of multiple rotary belt cooling granulators is necessary for a large-scale sulfur recovery device. As illustrated in Figure 3, annual throughput of sulfur granulating unit consisting of 8 rotary belt cooling granulators in parallel reaches 300,000 tons.

At present, maximum throughput of a rotary belt cooling granulator is 6,000kg/h; its utility conditions
and natural parameters are as follows:

Throughput: equal to or less than 6,000 kg/h

Particle size: diameter ranges from 3 to 6 mm; thickness ranges from 1 to 2 mm.

Particle shape: hemispherical

Feeding temperature: 130°C

Discharge temperature: 50-65°C

Moisture content of finished product: equal to or less than 0.5%

Bulk density: 1,200 kg/m³

Friability: equal to or less than 1.0%

Angle of repose: 30°

Yield rate: equal to or more than 95%

Utility:

(1) Power supply: 380V, 50Hz, 3-phase

(2) Gross power: 8KW

(3) Cooling water: Inlet temperature is equal to or less than 30°C; outlet temperature is equal to or less than 30°C; circulating water consumption is equal to or less than 20 m³/h.

(4) Dimension: 12950*2050*2060
Figure 3 Sketch Map of Technological Process of Sulfur Granulation Equipment with Annual Productivity of 300,000 tons

1. Sketch Map of Process

Heat filter  Liquid sulfur enters  a  Belt conveyor a
b  Belt conveyor b  Intermediate bin
Rotary belt cooling granulator  Liquid sulfur refluxes
Release agent  Water  Release liquid make-up tank
Cooling water enters  Cooling water returns

2. Air Cooling Tower Granulating Process
Air cooling granulating process: molten sulfur dropping from the top of the tower is distributed via nozzle; air is blown from the bottom of the tower to the top of the tower; liquid sulfur is cooled and solidified by rising air when it drops. Solid sulfur is collected on the bottom of the tower.

Air cooling granulating process is first called Outokumpu Oy Process which was first applied by Kokkola Sulfur Plant of Outokumpu Oy, Finland; air cooling granulating unit was set up in Kokkola and ceased to be used in 1977. Two granulating units applying air cooling granulating process were set up in Japan.

In addition to this process, other process suitable for mass production is called Polish Process; please see Figure 4 for principles of Polish process.

Air cooling granulating process: to store degassed liquid sulfur from sulfur recovery device in liquid sulfur tank; temperature of liquid sulfur should be kept between 130°C to 140°C in order to prevent liquid sulfur from freezing; therefore, temperature of liquid sulfur tank should be kept and liquid sulfur tank should be equipped with steam coil heater; low-pressure steam is used to heat the tank and keep its temperature.

Use liquid sulfur pump to pump liquid sulfur stored in liquid sulfur tank, convey it to the top of prilling tower at a constant speed and distribute it into the tower via a sprayer; cooling air enters from the bottom of the tower; seed crystal (sulfur granule) is kept at granulating area to accelerate the forming of crystal nucleus; when the granule drops to moving grizzly at the bottom of the tower, granule will be solidified. Sulfur granule produced via air cooling granulation process is 1 mm to 6 mm in diameter, 0.5% in moisture content, 1,100 kg/m³ in bulk density, 1.0% in friability and 25º in angle of repose.

Prilling tower is 30 m to 90 m in height and 3 m to 24 m in diameter, depending on throughput of a granulating unit. Polish Process was first applied in a pilot factory in Dobczyce, Poland in 1966 with annual throughput of 150,000 tons and then applied in a sulfur factory in Gdańśka with annual throughput of 500,000 tons; in the early 1980s, 5 sets of air cooling tower granulating units were used in Canada; annual throughput of these granulating units ranges from 350,000 tons to 1,200,000 tons, all of which used stand-alone prilling tower.

Air cooling tower granulation process doesn’t require water; as there are little rotating parts and operation performance is reliable, air cooling tower granulation process is suitable for mass production; however, heavy investment is required; as air cooling is required, so energy consumption is high.
3. Water Cooling Granulating Process

Water cooling granulating process: to enable liquid sulfur to be sprayed into or dropped into water tank or water tower and to be solidified and filtered; RIM process, Hessoil Virgin Island process, Devco Wet granulation process, Smth & Ardusi granulation process represents this kind of process.

Please see Figure 5 for working principle of RIM process; RIM process: to pump liquid sulfur with liquid sulfur pump into the top of 15.2m-high prilling tower and divide liquid sulfur into many liquid bundles via liquid sulfur distributor, and enable liquid bundle to flow into a water tank. Under the joint action of gravity and surface tension, liquid bundle forms granules which is conveyed to dewatering screen from the bottom of the tower; cooling water from prilling water can be reused.

At present, the most commonly used wet granulating process is Devco Wet granulating process.

Please see Figure 6 for Devco Wet Granulating Process: liquid sulfur from liquid sulfur recovery unit is conveyed to liquid sulfur tank after being filtered by sulfur filter. In addition, fine sulfur that can not be used as finished product (i.e. particle diameter can not meet the requirement after being screened by vibrating screen) is conveyed to sulfur melting pot; after being melted, sulfur is conveyed through molten sulfur pump to liquid sulfur tank. Temperature of liquid sulfur should be kept between 130°C to 140°C in order to prevent liquid sulfur from freezing; therefore, temperature of liquid sulfur tank should be kept and liquid sulfur tank should be equipped with steam coil heater; sulfur melting pot is equipped with steam coil heater in order to melt fine sulfur particles.

Use liquid sulfur pump to pump liquid sulfur stored in liquid sulfur tank and convey it to liquid sulfur distributor at a constant speed; a pipeline for returning liquid sulfur to liquid sulfur tank is set up in liquid sulfur conveying pipeline in order to implement production regulation; liquid sulfur conveying pipeline is equipped with steam jacket in order to prevent liquid sulfur from freezing at the pipeline.

Liquid sulfur is divided into many liquid bundles by liquid sulfur distributor and flown into a granulator (cooling tank); under the joint action of gravity and surface tension, liquid bundle forms granules which are conveyed to dewatering screen from the bottom of the cooling tank; cooling water from cooling tank can be reused.

Solid sulfur with a moisture content of 2% separated by vibrating screen (particle size meets the requirements) is conveyed to dryer (including fluidized bed unit, blower, fuel gas heater, inlet header box, exhaust header box, cyclone dust remover, wet scrubber, air pump, etc; notes: this dryer is an optional unit) via sulfur conveyor for rehandling or directly conveyed to sulfur weighing, packing and stacking system.

Fine sulfur particles separated by vibrating screen (fine sulfur particle can not be used as finished product
because its diameter cannot reach the requirement) is, together with process water, conveyed to fine separating tank in which finer sulfur particle is separated and conveyed to sulfur melting pot.

Finished product of sulfur directly contacts with cooling water; process water from process water tank exchanges heat through heat exchanger, in which temperature will drop from 53°C to 49°C; and then process water is conveyed to granulator (cooling tank), in which process water is partly returned to process water tank after overflowing from cooling tank and partly conveyed to fine separating tank after being separated by vibrating screen and then returned to process water tank after overflowing, and partly changed into vapor in cooling tank and then emitted to the atmosphere via blower. Losing process water is conveyed to process water tank through make-up water system. Process water can be reused.

Sulfureted hydrogen and sulfur vapor will emit in the process of forming solid sulfur. A blower is equipped to pump sulfureted hydrogen and sulfur vapor in order to keep environment clean.
Figure 6 Sketch Map of Devoc Wet Granulation Process
Water cooling granulating process requires water and finished product of sulfur can directly contact with cooling water; however, energy consumption is high. Water granulating process is economical and reliable for small lot production and lot production; water cooling granulating unit is not easily damaged, with low cost of production and maintenance.

Standard sulfur granule produced via water cooling granulating process is 1 to 6 mm in diameter, 2.0% in moisture content, 1,100 kg/m3 in bulk density, 3.0% in friability and 32º in angle of repose.

Devco wet granulating process does not require many granulating units, therefore featuring space saving. Please see Figure 7 for sketch map of layout of Devco wet granulating process.
4. Spray Granulating Process

Principle of spray granulating process: to spray and attach a layer of liquid sulfur to solid sulfur to further solidify solid sulfur and increase particle size. Attach a layer of liquid sulfur to solid sulfur to further solidify solid sulfur and continue to repeat this step until the particle size meets the requirement; this process uses water or air as coolant. Spray granulating process mainly includes Perlomatic Process, ProcorGX Process and Kaltenbach-Tharing Process.

Sulfur produced via spray granulating process is 1 to 6 mm in diameter, 0.5% in moisture content, 1,200 kg/m³ in bulk density, 1.0% in friability and 27º in angle of repose.

Maximum daily throughput of a product line applying spray granulation process has reached 1,100 tons; spray granulating process features space saving and high efficiency and is particularly suitable for campaign production of liquid sulfur.

1) Perlomatic Process:

Liquid sulfur is conveyed from the bottom of the equipment; under the action of strong air flow, liquid sulfur is sprayed towards a direction when it reaches larynx of venture tube. Under the action of air flow, solid sulfur at the top of venture tube shows fountain movement and is coated by molten sulfur. Under the action of strong air flow, particles at the center of fluidized bed rise to the upper side of the bed and then drop to all sides of the surface of the bed, therefore producing circulatory movement. As particles are being continuously coated by molten sulfur and cooled, they become bigger. When particle
size meets the requirements, particles will be conveyed to screen via overflow weir and stored; air is released from the top of the vessel which is just like an expansion tank where a majority of sulfur is separated prior to the release of air. Optimal temperature (usually 82°C) of fluidized bed is kept; this temperature is kept by keeping temperature of air entering the bed at 30°C.

Perlomatic Process is actually a fluidized bed spray granulating process; Perlomatic Process consumes more power and produces more exhaust than air cooling tower granulation process does. Please see Figure 8 for Perlomatic Process.

Figure 8 Sketch Map of Perlomatic Process

2) GX Tumbling Granulating Process:

Enersul Group is well-known for its sulfur forming service. Patented sulfur forming technology was first released in 1977, the basis of which is fertilizer granulation technology and TVA experiments on sulfur coating of urea; standard Procor Gx Process has been designed with a design forming capacity of 8 to 70 tons per hour; 13 sets of granulation units applying Procor Gx Process have been set up globally by the end of the 1990s, with total forming capacity of 2 million tons per year. Enersul Group
have sold GX Tumbling Granulating Units to Greece North Aegean Petroleum Company (NAPC), Qatar Petrochemical Company, Qatar Qatargas and Qatar Rasgas, Taiwan Formosa Petrochemical Corporation, SASOL in South Africa, South Pars1, South Pars2&3 in Iran by October 2002.

Sulfur granule produced by GX tumbling granulating unit is resistant to mechanical injury. GX tumbling process is a process of gradually increasing particle size; fine particles are coated and mingled, thus eliminating the effect of contraction and producing harder and defect-free granules with bigger bulk density (1220kg/m³ when the structure is loose; 1320kg/m³ when the structure is compact).

GXM1 granulating process and GXM2 granulating process are typical of GX tumbling granulating process.

Each GXM1 granulating unit can produce up to 1,100 tons of high quality sulfur granules per day and covers an area of 480m² (40m*12m), with investment of 35.46 million Yuan. Sulfur granule produced by GXM1 granulating unit has general performance of sulfur granule produced via GX tumbling granulating process; intermediate materials are conveyed via conveyor.

Please see Figure 9 for sketch map of Enersul GXM1 tumbling granulating process.

![Figure 9 Sketch Map of Enersul GXM1 Tumbling Granulating Unit](image)

Each GXM2 tumbling granulating unit can be designed to produce a daily output of 550 tons of granules and covers an area of 65 m² (10m*6.5m). Two GXM2 tumbling granulating units can produce up to 1,100 tons of sulfur granules per day and covers an area of 144m² (12m*12m), with total investment of 29 million Yuan. Sulfur granule produced by GXM2 granulating unit has general performance of sulfur granule produced via GX tumbling granulating process; intermediate materials are vertically conveyed; please see Figure 10 for sketch map of Enersul GXM2 tumbling granulating process.
Take Enersul GXM2 tumbling granulating process (please see Figure 12) as an example:

Liquid sulfur from sulfur recovery unit is stored in liquid sulfur tank (TK 101) which is divided into two parts by a partition board: part one is used as molten sulfur tank and part two as a true liquid sulfur tank. When liquid sulfur in molten sulfur tank reaches certain height, it will get across the partition board and enter into liquid sulfur tank. Temperature of liquid sulfur in liquid sulfur tank (TK 101) should be kept between 130°C and 140°C in order to prevent liquid sulfur from freezing. Therefore, temperature of two parts of liquid sulfur tank (TK 101) should be kept and they should be equipped with a set of steam coil heater.

Liquid sulfur in liquid sulfur tank is pumped to dual channel filter and then to tumbling granulating chamber (GD 101) at a constant speed by using liquid sulfur delivery pump (P 101). Liquid sulfur conveying pipeline is equipped with a pipeline through which liquid sulfur returns to liquid sulfur tank in order to exercise production control. Liquid sulfur conveying pipeline is equipped with vapor jacket in order to prevent liquid sulfur from freezing in the pipeline, the temperature of which is kept by using 0.5Mpa vapor.

When liquid sulfur is pumped to tumbling granulating chamber (GD 101), fine sulfur particles (seed particles) form continuously descending particle curtain under the action of partition board at inner wall of the chamber; these particles will be coated with a layer of liquid sulfur. Cooling and coating runs through the whole drum exchanger; each layer is cooled and solidified and coated with a
layer of liquid sulfur; this step is continuously repeated, in which particle size and weight is increased, until particle size meets the requirement. Particle diameter usually ranges from 1 mm to 6 mm (please see Figure 11). Then, particles are conveyed to vibrating molecular sieve (SE 101) for separation; particles meeting the requirements will be conveyed to the next working procedure (optional) by product conveyor (PC 101) for rehandling or be directly conveyed to sulfur weighing, packing and stacking system. Solid sulfur powder (particle size of which can not meet the requirements) screened by vibrating molecular sieve (SE 101) is recovered and conveyed to liquid sulfur tank (TK 101) for remelting. Liquid sulfur remelted from solid sulfur powder, together with liquid sulfur from sulfur recovery unit, will be pumped using liquid sulfur delivery pump (P 101) for use.

![Figure 11: Inner Chamber of Tumbling Cylinder](image)

Cooling water filtered by cooling water filter (F 101) is pumped using cooling water pump (P 102) to tumbling granulating chamber (GD 101). Heat in solidified sulfur will be vaporized by cooling water and be released from the chamber via air flow and discharged into the atmosphere after being separated by cyclone separator (CY 101).

As far as tumbling granulating process is concerned, proportion of seed particle, liquid sulfur and water must be strictly controlled with small scope of regulation. Therefore, tumbling granulating process is less flexible.
Figure 12 Sketch Map of GXM2 Sulfur Granulating Process
III. Comparison and Selection of Granulation Process

Each granulating process has its advantages and disadvantages. Selection of sulfur granulating process depends on industrial characteristics, sulfur recovery process and production scale. The most suitable sulfur granulating process should be chosen. Sulfur granulating process varies from petroleum industry, natural gas industry and coal chemical industry to smelting industry and power generation industry, and varies with production scale, modes of production and market structure. It is difficult to say that certain sulfur granulating process is suitable for all fields.

Based on the foregoing introduction and analysis of sulfur granulating processes, the author believes that, in addition to such normal indicators as energy consumption and investment, consideration should be given to the following three factors when choosing sulfur granulating process:

1. Throughput of sulfur granulating unit

2. Requirements for throughput fluctuation scope: raw materials may change over time; sulfur content of crude oil in particular may change significantly and market demand for liquid sulfur and solid sulfur may also change, which may cause a big difference between peak throughput and valley throughput. Therefore, consideration must be given to whether granulating unit can adapt to the change when we select a granulating process.

3. Actual requirements for quality indicators of finished product of sulfur granules: in terms of actual
requirements for application of finished product of sulfur granules, some industries (such as acid making industry) may have lower requirements for moisture content of granule sulfur. Therefore, water cooling granulating process is more competitive if it is accepted by users.

To sum up, characteristics of the foregoing granulating processes are summarized as follows:

Rotary belt cooling granulating process is still most extensively applied. As sulfur granule produced by rotary belt cooling granulating unit has a similar particle size distribution (3 to 6 mm; particle diameter of sulfur granule produced via other granulating processes ranges from 1 mm to 6 mm) and has uniform particle size without screening and returning materials. Cooling water comes into indirect contact with materials without discharging solid and liquid waste and exhaust. This process is simple and direct with lower investment and energy consumption, so it is the preferred process for small to intermediate capacity sulfur producers. However, handling capacity of sulfur recovery unit will increase with production expansion; this process is limited by throughput (maximum throughput: 6,000 kg/h) of stand-alone rotary belt cooling granulating unit and higher production levels can only be achieved by adding additional units covering larger footprint space, so larger-scale application of this process is limited. It is understood that Shell Canada Ltd set up a set of sulfur granulating unit with annual throughput of 1.6 million tons using this process between the late 1980s and the early 1990s, which consisted of 46 rotary steel belt cooling granulating units supplied by SANDVIK. This process was also applied to Tianjin Petrochemical 1-million-ton ethylene and sulfur recovery and granulating project undertaken by Nanjing Sunup Granulation Equipment Co., Ltd; this sulfur granulating unit with annual throughput of 300,000 tons consisting of 8 rotary steel belt cooling granulating units was set up and put into use in March 2008 and was the biggest sulfur granulating unit ever set up in China.

Air cooling tower granulation process requires heavier investment and air cooling and higher energy consumption and handling of more exhaust, so this process is being eliminated or maintained. Based on information available, three sets of sulfur granulating units applying air cooling granulation process (large-sized cooling tower) are being used. In recent years, there have been no reports of this process so far.

Water cooling granulating process requires higher energy consumption as sulfur granule comes into direct contact with cooling water. Therefore, water cooling granulating process is economical and reliable for small lot and lot production; water cooling granulating unit is not easily damaged. Costs for production and maintenance are kept low. Main disadvantages are as follows: as sulfur granules come into direct contact with process water and moisture content of sulfur granule is around 2.0%, sulfur granules fail to meet the requirements of Sulfur for Industrial Use and Its Testing Methods (GB2449-92) (requirements of moisture content: moisture content of superior quality sulfur granule must be equal to or less than 0.10%; moisture content of first-class sulfur granule must be equal to or less than 0.50%; moisture content of passed-quality sulfur granule must be equal to or less than 1.00%). Therefore, finished product of sulfur granule must be dried; moisture content of finished product of sulfur granule is around 1.00%, the moisture content of passed-quality
sulfur granule. In recent years, more than 10 sets of sulfur granulating units have applied water cooling granulating process. This process with daily maximum throughput of 2,000 tons is quite effective and relatively suitable for large-scale production line if quality requirements are not strict; therefore, when moisture content requirements of sulfur granule are not so strict, foreign factories often select this process because investment and operating costs are kept low.

Spray granulating process falls into two categories: fluidized bed spray granulating process and tumbling spray granulating process. Fluidized bed spray granulating process consumes more energy and produces more exhaust than air cooling granulating process does. Although investment in fluidized bed spray granulating process is comparatively low, the author does not think that the application of this process will have a promising prospect. Tumbling spray granulating process represents technical development direction of spray granulation and is suitable for larger-scale production with comparatively fixed throughput. However, proportion of seed particle, liquid sulfur and water must be strictly controlled with small scope of regulation and this process is thus less flexible and not suitable for granulation with bigger fluctuation of throughput. In addition, investment in this process is comparatively high. It is understood that this process have been applied to natural gas desulphurization project of China Petroleum & Chemical Corp. Puguang Oil and Gas Field and complete sets of tumbling spray granulating units have been introduced.

IV. Conclusion and Suggestion

To sum up, consideration should be given to technical and economic factors, production management & scheduling, energy consumption & operating costs, and the state of China. The following suggestions and advices for selection of sulfur granulating process are offered:

1. It is suggested that rotary belt cooling granulating process should be suitable for sulfur granulating unit with annual throughput of 800,000 tons and bigger fluctuation of throughput or with annual throughput of 500,000 tons.

2. It is suggested that water cooling granulating process should be suitable for sulfur granulating unit with less strict moisture requirements of sulfur granule and annual throughput of more than 300,000 tons. Therefore, the author suggests that it should be necessary to modify the moisture content provision of Sulfur for Industrial Use and Its Testing Methods (GB2449-92) and moisture content requirements should be relaxed.

3. It is suggested that tumbling spray granulating process should be suitable for sulfur granulating unit with annual throughput of more than 500,000 tons and smaller fluctuation of throughput.