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Urea particle coating for controlled release by using DCPD modified sulfur

Yong-Hui Liu, Ting-Jie Wang *, Liang Qin, Yong Jin

Department of Chemical Engineering, Tsinghua University, Beijing 100084, China

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Abstract

The shell of sulfur coated urea was easily cracked due to sulfur being friable. Sulfur was modified with dicyclopentadiene (DCPD) to increase its strength and abrasion resistance. SEM images showed that the micro-structure of modified sulfur was denser and more uniform than pure sulfur. The strength of modified sulfur increased with the DCPD/S ratio. Experiments of urea particle coating with sulfur and modified sulfur were carried out in a fluidized bed coater. The shell of coated urea particles with modified sulfur was more compact than that with pure sulfur. The modification retarded the sulfur phase transformation from monoclinic to orthorhombic, avoiding the crack formation in the coating shell of sulfur. The modified sulfur coated urea particles can be produced with thinner shell and higher strength, and had better controlled release properties.

Keywords: Coating; Urea; Sulfur; Modification; Control release

1. Introduction

Sulfur coated urea (SCU) has been produced for about 40 years. Many works have reported the improvement of the coating quality [1,2]. Due to that sulfur is friable, the coated shell cracks easily, and the shell is even peeled from the particle surface during transportation, stockpile storage, and fertilization. Wax and wax-like materials were usually coated on the outer surface of the sulfur coated urea to seal the flaws to reduce the release rate. However, these have a high cost, and the wax coating made the product adhesive so that additional treatments have to be set in the flowsheet for improving the fluidity of SCU particles.

Sulfur was plasticized by adding modifiers in sulfur containing composites for road repair, road-making material and concrete in building construction [3–6]. Most modifiers reported in literature were polymeric polysulfides or, alternatively, substances which react with sulfur, such as mercaptan and unsaturated hydrocarbon, to give *in situ* formation of polymeric polysulfides. Blight et al. [7] studied modifying sulfur with dicyclopentadiene (DCPD) and styrene, and showed that the modifiers can stop or reduce sulfur embrittlement. Bordoloi and Pearce [8] researched the viscosity of sulfur-DCPD solutions varied with reaction time and compositions, and showed that the viscosity increased exponentially as the time and DCPD quantity increased. A kind of sulfur-plasticizing reagent with the formula A–R–S_x–R–A was reported [9], where each *R* is a hydrocarbon radical having up to about 10 carbon atoms, at least one *A* is hydroxyl or carboxyl and *x* is an integer from 2 to 5. This polysulfide modifier was prepared by the reaction of sulfur with a mercapto compound selected from mercapto acids and mercapto alcohol in the presence of basic catalysts such as amines and ammonia. The viscosity of modified sulfur could be reduced by adding persulfides with A–R–S_x–R–A structure. In addition, a slow release particulate fertilizer product in which the fertilizer particles were encapsulated with a plasticized sulfur coating, where the plasticized sulfur was prepared by adding a certain amount of a substituted symmetrical dialkyl polysulfide plasticizer to sulfur has been reported [10].

In this paper, DCPD, a product from petroleum cracking, was used as the modifier to prepare modified sulfur as the coating material for producing controlled release urea. The properties of the modified sulfur and the sulfur coated urea particles were investigated.

2. Experimental

2.1. Materials

Commercial urea particles with size in the range of 2~4 mm and 1335 kg/m^3 in density were used in the experiments, which were produced by the Ningxia Petrochemical Company of

* Corresponding author. Tel.: +86 10 62788993; fax: +86 10 62772051.
E-mail address: wangtj@mail.tsinghua.edu.cn (T.-J. Wang).

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PetroChina Co. Ltd. Dicyclopentadiene was a commercial product with purity 94.0% from the Hangzhou Yangli Petrochemical Co. Ltd. Sulfur was a commercial product with purity 99.9%.

2.2. Sulfur modification

A known amount of DCPD and commercial sulfur were mixed uniformly in a beaker at 145 °C controlled with an oil bath. After a specified time of reaction, the modified sulfur was obtained. The reaction time is in the range of 1–6h.

Experiments showed that the viscosity of the molten modified sulfur significantly increased with the DCPD fraction and the reaction time. Bordoloi and Pearce proposed a viscosity expression for modified sulfur with DCPD at 140 °C [8],

$$\eta = 19.46 \exp\left(11.33X^{1.78}t\right), \quad [X = 0 - 0.4] \tag{1}$$

where η is the viscosity of molten modified sulfur, cP; X is the mole fraction of DCPD; t is the reaction time, h. Considering that too high a viscosity is not suitable for the spraying coating process, therefore, based on the exploratory experiments, the DCPD fraction was set in the range of 0–10% sulfur by weight.

2.3. Fluidized bed coater and coating procedures

A fluidized bed coater, shown in the Fig. 1, was used for the coating of the urea particles. The fluidized bed was made of an organic glass column 150 mm in diameter and was widened in the upper section at 300 mm to restrict particle entrainment. A spraying nozzle of an air-atomized nozzle was centrally set above the fluidized bed.

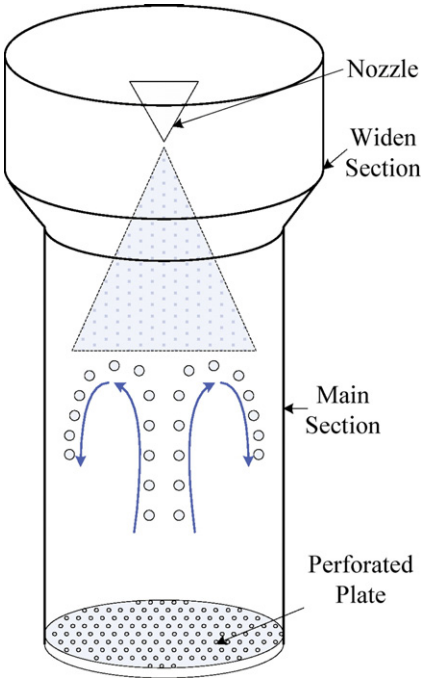

Fig. 1. Schematic diagram of the coating process in a fluidized bed.

Table 1
Operation parameters of coating process in a fluidized bed

Superficial gas velocity, m/s	Air pressure, MPa	Air flow rate, L/min	Molten sulfur flow rate, mL/min	Molten sulfur temperature, °C	Temperature in coater, °C
2	0.2	25	30	145	60

Urea particles of 1kg in weight were put in the fluidized bed, and the bed was fluidized at a superficial gas velocity of 2.0 m/s. The molten coating material under a pressure of 0.2 MPa was atomized with compressed air, and sprayed onto the urea particles in the fluidized bed for coating. The operation parameters in the coating process were listed in Table 1. After spraying a set quantity of coating material, the particles were taken out for analysis.

2.4. Measurement of strength and abrasion resistance

In order to examine the properties of modified sulfur as a coating material, the compressive strength of the particles prepared with the modified sulfur was measured. Modified

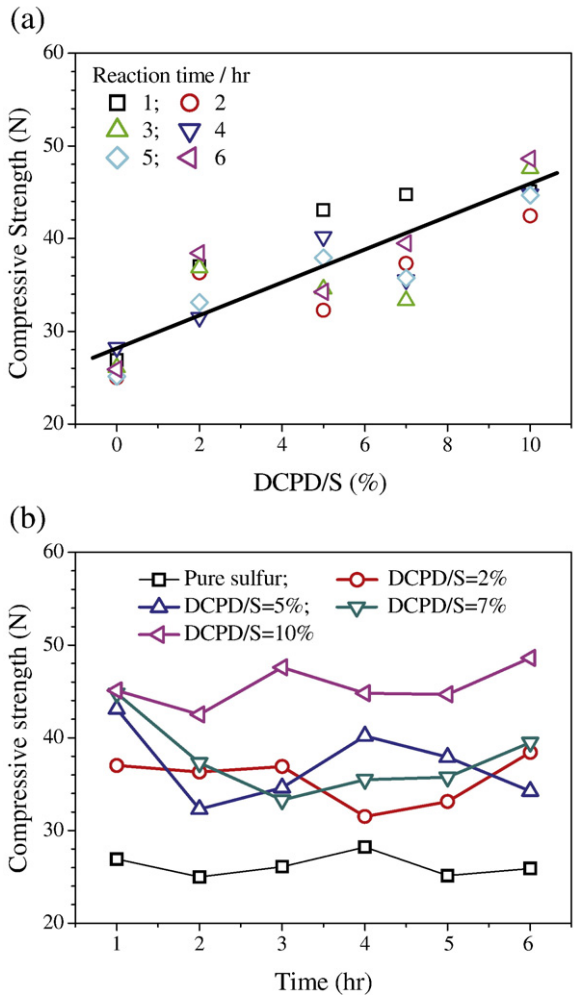

Fig. 2. Compressive strength of sulfur/modified sulfur granules versus DCPD/S ratio and reaction time.