



Effects of polymer coated urea and sulfur fertilization on yield, nitrogen use efficiency and leaf senescence of cotton

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ABSTRACT

Nitrogen (N) and sulfur (S) fertilization play important roles for improving cotton yield, but no studies have been implemented to explain their interaction on yield, nitrogen use efficiency and physiological characteristics of cotton. In order to investigate the interaction effects of polymer coated urea (PCU) and S fertilization on the contents of inorganic N and available S, enzymes activities of leaves and yield of cotton, the field experiment with different types of N fertilizers and S rates was carried out in 2014 and 2015. The experiment consisted of two N fertilizer types including PCU and common urea fertilizer (Urea) in combination with three S rates (0, 60 and 120 kg ha⁻¹) in the split-plot design, where the types of N fertilizer were the main plot and S rates were the subplots. The results indicated that the N release characteristic of PCU in field condition was closely matched to the N requirements of cotton, the contents of soil nitrate nitrogen (NO₃⁻-N) and ammonium nitrogen (NH₄⁺-N) were significantly increased from the first bloom stage to the initial boll-opening stage by using PCU compared with urea. And the content of available S was significantly increased in full boll setting stage. Meanwhile, the number of bolls and lint yields of PCU were 7.03–8.91% and 5.54–11.17% higher than urea treatments. Lint yields were also increased 3.77–9.26% by S fertilization, evidencing a clear interaction between N and S, but no significant difference was observed between S60 and S120 treatments. In addition, the N apparent recovery use efficiency (RUE) and agronomic use efficiency (AUE) were increased, fiber length and strength were improved, the nitrate reductase and peroxidase activities and photosynthetic rates (Pn) were enhanced by PCU and S fertilization. However, the lint percentage, micronaire and fiber elongation were neither affected by the type of N fertilizers and S rates, nor by their interaction. Consequently, the application of PCU combined with 60 kg ha⁻¹ sulfur fertilizer on cotton could not only increase the yield and nitrogen use efficiency but also improve the fiber quality and physiological properties of leaves.

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1. Introduction

Cotton is one of the important cash crops and a fiber crop most widely grown in the world (Ali, 2015). Nitrogen is an essential element that is required most consistently and in larger amounts than other nutrients, which has contributed greatly to cotton production (Chen et al., 2010; Dong et al., 2012). Sufficient N initially supports the rapid development of roots and leaves, increases cotton biomass accumulation and yield formation, and also needed for formatting chlorophyll (Singh et al., 2010; Silvertooth et al., 2011). Usually common urea fertilization added in three different times correspond to the phases of plant growth (Yang et al., 2011) to match the dynamic N demand in cotton growth cycle. However,

Abbreviations: PCU, polymer coated urea; NUE, nitrogen use efficiency; NR, nitrate reductase; GS, glutamine synthetase; SOD, superoxide dismutase; POD, peroxidase; MDA, malonyldialdehyde.

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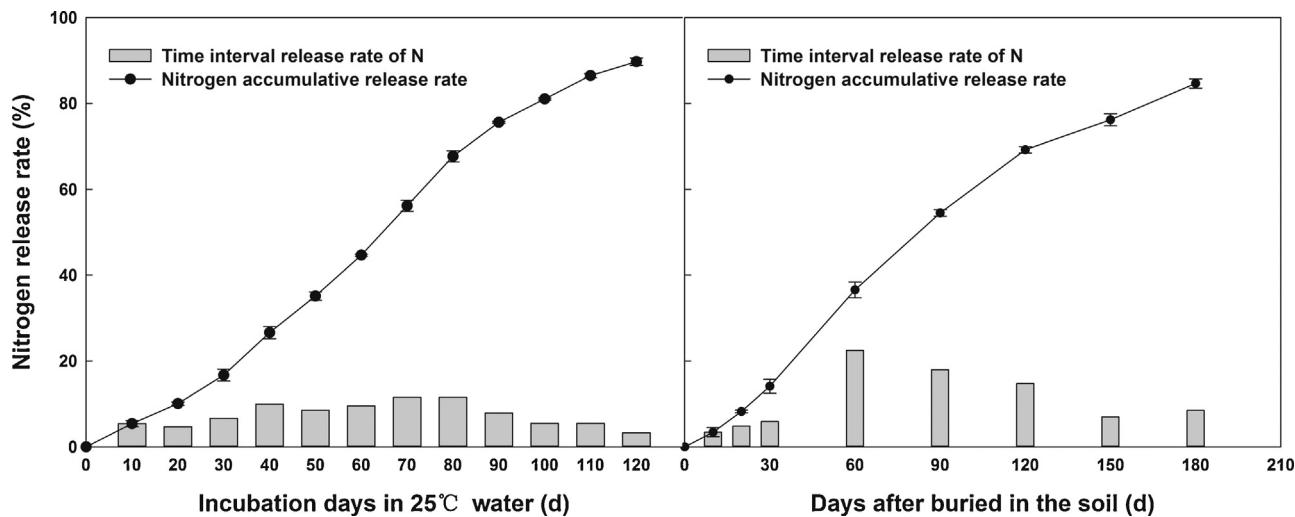


Fig. 1. Nitrogen release rate for period of time and accumulative release rate of PCU in water and field condition. Error bars represent standard error from mean ($n=3$).

the application of too much common nitrogen-containing fertilizer is not economical, because it has the negative impacts on both environmental and economic sides. Moreover, the large amounts of external N inputs and the improper ways of N fertilization resulted in serious soil degradation, a low nitrogen use efficiency (NUE), greenhouse gases and groundwater pollution risk (Guo et al., 2010; Spiertz, 2009; Zhang et al., 2012b). In addition, the split application of urea is more costly than one-time fertilization, and reducing the profit from cotton planting (Yang et al., 2012a). Thus, it is necessary to optimize N fertilizer inputs both to meet crop requirement and to reduce environmental pollution. To this end, the controlled release urea fertilizers are produced to reduce cost, to facilitate of application, and to achieve higher NUE (Yang et al., 2012b). Several slow and controlled release fertilizers have been developed, including polymer coated urea (PCU), sulfur coated urea, polymer coating of sulfur-coated urea and urea formaldehyde. Some researchers found that the application of controlled release fertilizer promote the growth of cotton significantly and enhance the N supply in the whole growth periods (Wang et al., 2013).

Sulfur (S) is another important factor influencing cotton yield, the plant growth and development. It is found in cysteine, amino acids that make up proteins, and activates certain enzyme systems (Najafian and Zahedifar, 2015), and S is also an essential constituent of enzymes involved in N metabolism like nitrate reductase (NR)

and nitrite reductase (Swamy et al., 2005). A positive interaction between N and S in increasing crop biomass and yield is observed by Salvagiotti and Miralles (2008), and S fertilization improves NUE in wheat by increasing nitrogen uptake (Salvagiotti et al., 2009). Likewise, an increase in leaf photosynthesis is expected when S supply is increased (Terry, 1976). Geng et al. (2015) also reported that polymer coating of sulfur-coated urea achieved more cotton yield compared with polymer coated urea, it may because S fertilization increased the plant biomass. However, sulfur deficiencies are often observed in agronomic systems due to the use of highly concentrated fertilizers containing little or no S element and increased crop yields, which result in more S removal from the soil (Chen et al., 2005). An insufficient S supply can affect yield and quality of the crops, caused by the S requirement for protein and enzyme synthesis (Scherer, 2001). Consequently, more attention should be paid to S requirements of cotton and other cotton-producing states where S deficiencies may occur (Yin et al., 2011).

Although much effort has been made to explore the effects of N fertilization on cotton growth and its physiological components, there is a noteworthy lack of information about the S effects and especially on the interaction PCU × S in relation to the physiological attributes that determine NUE and yield of cotton. The objective of this study was to determine the influence of PCU and S supply and their interaction on (i) contents of nitrate nitrogen (NO_3^- -N),

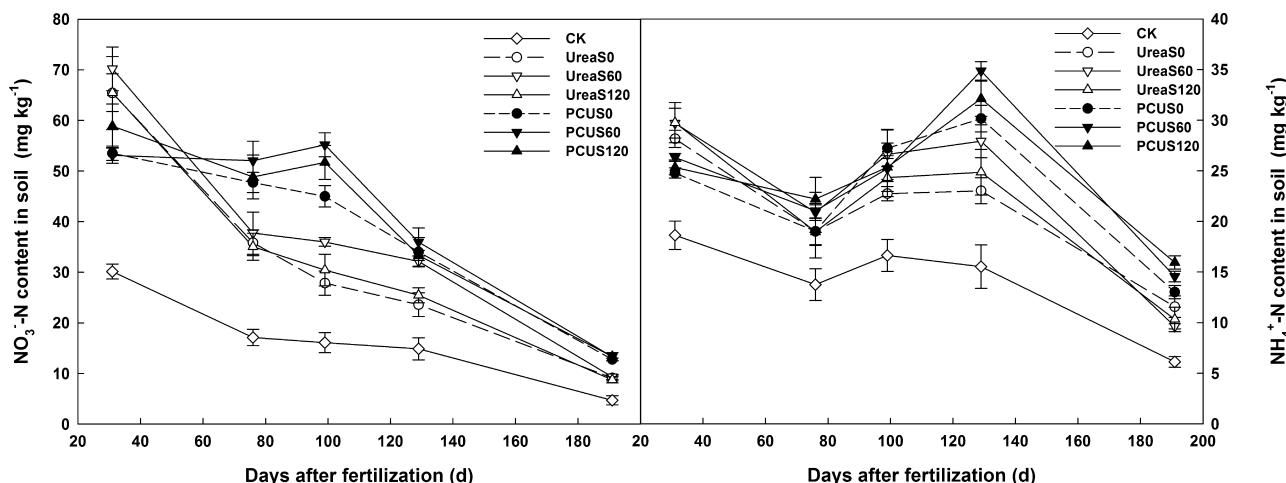


Fig. 2. Changes of NO_3^- -N and NH_4^+ -N content at different ontogenetic stages under different treatments. Error bars represent standard error from mean ($n=3$).

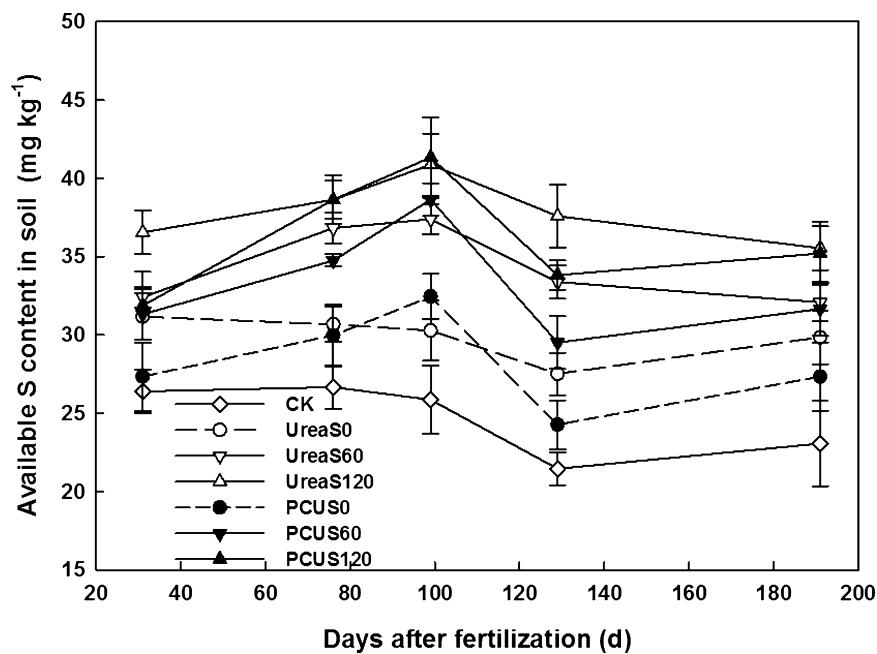


Fig. 3. Changes of available S content at different ontogenetic stages under different treatments. Error bars represent standard error from mean ($n=3$).

Table 1

Chlorophyll content (SPAD values) and ANOVA for the effects of N, S and their interaction effects on cotton at different ontogenetic stages.

Treatment	Chlorophyll content (SPAD value)				
	Squaring stage	First bloom	Full boll setting	Initial boll-opening	Full boll-opening
Types of N fertilizers					
PCU	65.99 a	56.62 a	67.23 a	37.71 a	30.77 a
Urea	63.59 b	55.37 b	63.18 b	35.64 b	27.46 b
S fertilizer rates					
S0	62.44 b	55.07 b	62.22 b	34.83 b	26.58 b
S60	67.37 a	57.39 a	68.43 a	38.47 a	31.36 a
S120	64.57 b	55.53 b	64.97 b	36.73 ab	29.46 a
N × S interaction					
CK	55.32 d	47.34 c	52.38 d	28.46 c	20.25 d
PCU × S0	63.80 b	55.72 ab	65.50 b	35.35 b	27.90 b
PCU × S60	68.73 a	57.62 a	69.03 a	39.58 a	33.08 a
PCU × S120	65.43 ab	56.53 a	67.17 a	38.19 a	31.33 a
Urea × S0	61.07 c	54.41 b	58.93 c	34.31 b	25.25 c
Urea × S60	66.00 a	57.17 a	67.83 a	37.36 a	29.63 ab
Urea × S120	63.70 b	54.52 b	62.77 bc	35.26 b	27.50 b
Source of variance					
N	0.0351	0.0001	0.0004	0.0106	0.0228
S	0.0087	0.0003	0.0002	0.0003	0.1752
N × S	0.8852	0.0484	0.0368	0.0477	0.1059

Note: PCU-polymer coated urea, Urea-common urea fertilizer. Means followed by a same lowercase letter in the same column was not significantly different by Duncan's test ($P<0.05$).

ammonium nitrogen ($\text{NH}_4^+ \text{-N}$) and available S in soil; (ii) changes of SPAD chlorophyll meter reading and photosynthesis of cotton leaves; (iii) enzyme activities of cotton leaves; and (iv) cotton yield, fiber quality and the NUE under field conditions in the north China plain.

2. Material and methods

2.1. Experimental materials

A field study was conducted in 2014 and 2015 at Hengshui district ($N 37^\circ 32'14''$; $E 115^\circ 28'59''$), Hebei Province of China with the cotton cultivar Guoxin3. The climate of the experimental area is temperate and monsoonal. The soil was silt loam (12.74% clay, 9.03% sand and 78.23% silt) with pH 8.18, soil total N and organic matter content was 0.95 and 14.6 g kg^{-1} , $\text{NO}_3^- \text{-N}$, $\text{NH}_4^+ \text{-N}$,

available P, available K, and available S was 30.21, 24.09, 42.93, 124.20 and 26.01 mg kg^{-1} , respectively.

The conventional fertilizer used was urea (46% N) as N fertilizer, common potassium chloride (60% K_2O) as K fertilizer, potassium dihydrogen phosphate (52% P_2O_5 and 34.02% K_2O), S granule (98% S) as S fertilizer. The polymer coated urea (PCU, 42% N, made by Kingenta Ecological Engineering Co., Ltd., Shandong, China) was used as controlled release urea for cotton. The N release longevity of PCU in water of 25°C was about 4 months.

2.2. Experimental design

A split-plot design with triple replications was used for the study. The main plots were assigned the type of N fertilizers (180 kg ha^{-1} polymer coated urea: PCU and common urea: Urea), while sulfur fertilizer rates (0 (S0), 60 (S60) and 120 (S120) kg ha^{-1})

Table 2

Enzymes activities and ANOVA for the effects of N, S and their interaction effects on cotton leaves.

Treatment	NR($\mu\text{gg}^{-1} \text{h}^{-1}$, FW)	GS(Ug^{-1} , FW)	SOD(U mg^{-1} Protein)	POD(U mg^{-1} Protein)	MDA($\mu\text{mol g}^{-1}$, FW)
Types of N fertilizers					
PCU	33.09 a	2.31 a	230.78 a	513.25 a	30.54 b
Urea	29.76 b	1.98 b	224.51 b	487.27 b	35.65 a
S fertilizer rates					
S0	28.43 b	1.87 b	222.92 b	497.17 a	34.66 a
S60	33.51 a	2.43 a	232.09 a	503.08 a	32.14 b
S120	32.34 a	2.14 a	227.94 b	500.52 a	32.50 b
N × S interaction					
CK	24.61 c	1.04 d	192.51 c	412.36 c	38.31 a
PCU × S0	30.25 b	1.97 b	226.21 ab	511.95 a	31.64 bc
PCU × S60	35.30 a	2.71 a	235.70 a	513.89 a	29.08 c
PCU × S120	33.71 ab	2.27 a	230.44 a	513.91 a	30.91 c
Urea × S0	26.60 c	1.78 c	219.63 b	482.38 b	37.68 a
Urea × S60	31.71 b	2.15 ab	228.48 ab	492.27 b	35.20 b
Urea × S120	30.96 b	2.01 ab	225.43 ab	487.14 b	34.08 b
Source of variance					
N	0.0030	0.0003	0.0021	0.0015	<0.0001
S	0.0043	0.0426	0.0108	0.6938	0.0293
N × S	0.0047	0.4695	0.1519	0.8408	0.0178

Note: PCU-polymer coated urea, Urea-common urea fertilizer, NR-nitrate reductase, GS-glutamine synthetase, SOD-superoxide dismutase, POD-peroxidase, MDA-malonyldialdehyde. Means followed by a same lowercase letter in the same column was not significantly different by Duncan's test ($P < 0.05$).

Table 3

Yield and its components and ANOVA for the effects of N, S and their interaction effects on cotton.

Treatment	2014					2015				
	No. bolls ($\times 10^5 \text{ ha}^{-1}$)	Boll weight (g)	Seed cotton yield (kg ha^{-1})	Lint percentage (%)	Lint yield (kg ha^{-1})	No. bolls ($\times 10^5 \text{ ha}^{-1}$)	Boll weight (g)	Seed cotton yield (kg ha^{-1})	Lint percentage (%)	Lint yield (kg ha^{-1})
Types of N fertilizers										
PCU	12.29 a	5.6 a	6779.0 a	44.2 a	2998.6 a	11.1 a	6.2 a	6828.7 a	45.1 a	3108.2 a
Urea	11.36 b	5.7 a	6449.7 b	44.1 a	2841.3 b	10.2 b	5.8 a	6222.2 b	45.6 a	2795.8 b
S fertilizer rates										
S0	11.33 b	5.7 a	6505.8 b	43.4 a	2821.2 b	10.0 b	5.8 a	6185.5 b	45.5 a	2796.2 b
S60	12.22 a	5.6 a	6734.0 a	44.7 a	3011.3 a	10.9 a	6.2 a	6723.8 a	45.3 a	3055.2 a
S120	11.88 ab	5.7 a	6603.3 ab	44.3 a	2927.5 a	11.1 a	6.0 a	6667.0 a	45.1 a	3004.7 a
N × S interaction										
CK	10.0 c	5.7 a	5709.9 d	42.7 b	2435.5 d	8.9 b	5.0 c	4455.0 c	44.4 b	1976.6 d
PCU × S0	11.8 ab	5.5 a	6616.6 b	43.7 ab	2893.2 b	10.7 a	6.0 ab	6368.4 b	46.9 a	2984.5 ab
PCU × S60	12.8 a	5.5 a	6993.7 a	44.8 a	3132.3 a	11.3 a	6.3 a	7091.1 a	45.2 ab	3202.6 a
PCU × S120	12.2 ab	5.8 a	6726.6 a	44.2 a	2970.4 b	11.4 a	6.2 ab	7024.5 a	44.7 ab	3137.6 a
Urea × S0	10.9 bc	5.9 a	6395.0 c	43.0 b	2749.1 c	9.3 b	5.6 bc	6002.6 b	44.1 b	2607.9 c
Urea × S60	11.6 ab	5.6 a	6474.3 bc	44.6 a	2890.2 b	10.5 a	6.1 ab	6354.6 b	45.5 ab	2907.8 b
Urea × S120	11.6 ab	5.6 a	6479.9 bc	44.5 a	2884.6 b	10.8 a	5.8 ab	6309.3 b	45.6 ab	2871.8 bc
Source of variance										
N	0.0051	0.1142	0.0002	0.6102	0.0010	0.0091	0.0481	0.0005	0.4069	0.0001
S	0.0165	0.2537	0.0171	0.0267	0.0036	0.0179	0.2755	0.0071	0.8799	0.0037
N × S	0.0358	0.0144	0.0173	0.4346	0.0408	0.0424	0.9012	0.0344	0.0859	0.0452

Note: PCU-polymer coated urea, Urea-common urea fertilizer. Means followed by a same lowercase letter in the same column was not significantly different by Duncan's test in the same year ($P < 0.05$).

were assigned to the subplot, and the treatment with no N and S fertilization was as the control (CK). The PCU were used as basal fertilization one time before sowing seeds, while urea used as twice-split fertilization, 50% at preplant and 50% at the first bloom stage. Meanwhile, the S fertilizer was also used as basal fertilization once before sowing seeds. Each main plot was 5 m wide and 18 m long, and each subplot was 5 m wide and 6 m long. There were 24 rows of cotton in each main plot and 8 rows per subplot with a row space of 70 cm and a density of 45,000 plants ha^{-1} . All plots received a basal application rate of 150 kg ha^{-1} P_2O_5 and 180 kg ha^{-1} K_2O based on local practice.

2.3. Sampling and measurement

2.3.1. Measurement of the N content and longevity of PCU

The N content and release rate of PCU in water were determined by the method of "State Standard of the People's Republic of China-Slow Release Fertilizer" (Liu et al., 2009), that 10 g PCU with three replicates were placed in a glass bottle containing 200 ml distilled

water, and then keep in a constant temperature incubator at 25 °C. The released N from PCU was determined using Kjeldahl method, and the solution samples were collected every 10 days until the accumulative N release rate of PCU was more than 80%. For the field method, the N cumulative release rates were measured by a weight loss method (Wilson et al., 2009). There were 30 mesh bags (10 g PCU prills were contained in each bag) were buried in the soil before planting cotton in 2015, and 3 bags were picked up every 10 days in the first month, and then 3 bags were collected every 30 days. The loss of weight was assumed as the rate of N release from PCU.

2.3.2. Soil sampling and analysis

The cotton seeds were sowed in April 23 of each year, and the soil samples in 0–20 cm depth from each plot were collected in seedling stage, first bloom stage, full boll setting stage, initial boll-opening stage and maturing stage in the days of 31, 76, 99, 129 and 191 after fertilization in 2015. The fresh soil samples were analyzed NH_4^+ -N and NO_3^- -N (extracted with 0.01 M CaCl_2) concentrations using

Table 4

Main effects of the type of N fertilizers, S rates and their interaction on fiber quality.

Treatment	Fiber length (mm)	Fiber uniformity(%)	Micronaire	Fiber elongation(%)	Fiber strength(cN tex ⁻¹)
Types of N fertilizers					
PCU	31.38 a	86.59 a	4.47 a	7.87 a	30.49 a
Urea	30.01 b	85.99 b	4.38 a	7.81 a	29.38 b
S fertilizer rates					
S0	29.60 b	85.48 a	4.37 a	7.85 a	28.90 b
S60	31.88 a	86.93 a	4.40 a	7.87 a	30.83 a
S120	30.61 ab	86.45 a	4.50 a	7.80 a	30.08 a
N × S interaction					
CK	26.67 d	82.77 d	4.46 ab	7.83 a	27.80 c
PCU × S0	30.59 b	85.97 b	4.48 ab	7.83 a	29.63 b
PCU × S60	32.59 a	87.30 a	4.47 ab	7.87 a	31.22 a
PCU × S120	30.96 ab	86.50 ab	4.45 ab	7.90 a	30.63 ab
Urea × S0	28.61 c	85.00 c	4.29 b	7.87 a	28.17 c
Urea × S60	31.15 ab	86.57 ab	4.32 b	7.87 a	30.43 ab
Urea × S120	30.26 bc	86.40 ab	4.54 a	7.70 a	29.53 b
Source of variance					
N	0.0296	0.0429	0.1428	0.3175	0.0029
S	0.0143	0.0642	0.2236	0.5763	0.0011
N × S	0.0411	0.0386	0.1135	0.2036	0.5947

Note: PCU-polymer coated urea, Urea-common urea fertilizer. Means followed by a same lowercase letter in the same column was not significantly different by Duncan's test ($P < 0.05$).

the AA3-A001-02E Auto-analyzer (Bran-Luebbe, Germany) within 48 h after collected. And then the soil samples was air-dried, and ground pass through 2 mm sieve to measure the content of available S based on the precipitation of sulfate as BaSO₄ which extracted the soils with 0.05 M CaCl₂ (Kowalenko and Lowe, 1975).

2.3.3. Physiological characteristics determinations

Leaf gas exchange was measured by using a LI-6400XT portable photosynthesis system (LI-Cor, Lincoln, NE, USA) from 9:00 to 11:00 h under the standardized climatic conditions in each growth stage in 2015. The 3rd fully expanded young leaf that on the main-stem from terminal was taken to analysis of net photosynthetic rate (Pn). Meanwhile, the chlorophyll content was measured with a chlorophyll meter (SPAD-502; Minolta, Tokyo, Japan).

At full boll setting stage in 2015, 4 pieces of mature and fresh leaves from 4 successive plants were picked to laboratory. To measure antioxidant enzymes activities, 500 mg of fresh leaf tissue was homogenized using a chilled mortar and pestle in 50 mM phosphate extraction buffer (pH 7.0) and centrifuged at 10,000 rpm for 20 min at 4 °C. The supernatant was to assay the following antioxidant enzymes. The activity of superoxide dismutase (SOD) was assayed by measuring its ability to inhibit the photoreduction of nitro blue tetrazolium (NBT) using the method of Song et al. (2009); the peroxidase (POD) reaction was initiated by addition of H₂O₂, and the changes in absorbance of the reaction solution at 460 nm were determined every 30 s. Lipid peroxidation was estimated by measuring malonyldialdehyde (MDA) content, as described by Dhindsa et al. (1981) that the absorbance of the supernatant was measured at 450 and 532 nm and the values were corrected for non-specific turbidity by subtracting the absorbance at 600 nm. Nitrate reductase (NR) activity was measured in fully expanded leaves and placed in the buffer solution for in vivo NR activity assay based on the method of Aslam et al. (2001). Activity of glutamine synthetase (GS) was determined as described by Lea et al. (1990).

2.3.4. Yield and NUE

To measure cotton yield, a 5 m × 3 m area covering four central rows was picked up for 6 times and weighed after drying of each subplot in both years. All the bolls were recorded as the boll numbers, and 100 bolls were picked randomly at each picking and oven dried to calculate the average boll weight. Lint percent and lint yield was calculated after ginning. Then, the fiber samples were sent to cotton quality supervision and inspection center (Henan) for high

volume instrumentation determination of fiber quality parameters. The micronaire reading, length, strength, length uniformity index, and fiber elongation were determined for each sample.

Five successive plants above the roots from each subplot were sampled in 2015, separated them into vegetative organs (stem, leaves and branches), reproductive organs (buds, flowers and bolls), and enveloped them separately. Then, the samples were put into an electric oven for a quick cell killing at 105 °C for 30 min and drying at 75 °C to a constant weight before weighed. The total N concentration of plant was determined by H₂SO₄-H₂O₂ digestion and the micro-Kjeldahl procedure (Douglas et al., 1980). Above-ground plant N uptake was calculated from the sum of the dry matter and N concentration of the different plant parts. Apparent recovery use efficiency (RUE) and agronomic use efficiency (AUE) of nitrogen were calculated by the following formulas according to Devkota et al. (2013).

$$\text{RUE}(\%) = \frac{\text{N uptake at N applied plot} - \text{N uptake at N0 plot}}{\text{N rate in N applied plot}} \times 100\%$$

$$\text{AUE} = \frac{\text{Yield at N applied plot} - \text{Yield at N0plot}}{\text{N rate in N appliedplot}}$$

2.4. Statistical analysis

Data was statistically analyzed for analysis of variance as a split-plot factorial design with three replications. Two-way analysis of variation (ANOVA) was performed to determine the effect of the PCU, S and their interaction on yield, fiber quality, SPAD values and enzymes activities. One-way analysis of variance was performed to assess the significant differences of NH₄⁺-N, NO₃⁻-N, available S, Pn rate and NUE between different treatments. Analysis of variance and mean separation tests (Duncan's multiple range test, at the 5% probability level) was performed using Statistical Analysis System package version 9.2 (2010, SAS Institute Cary, NC). Means and standard error values were assessed to assemble graphs using the SigmaPlot software version 10 (MMIV Systat Software, Inc., San Jose, CA).

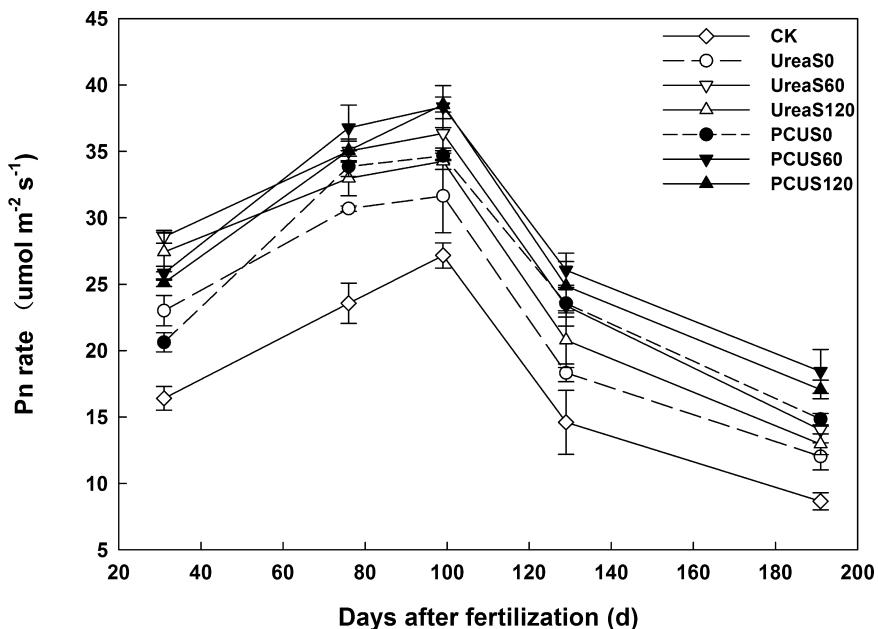


Fig. 4. Changes of Pn rate at different ontogenetic stages under different treatments. Error bars represent standard error from mean ($n=3$).

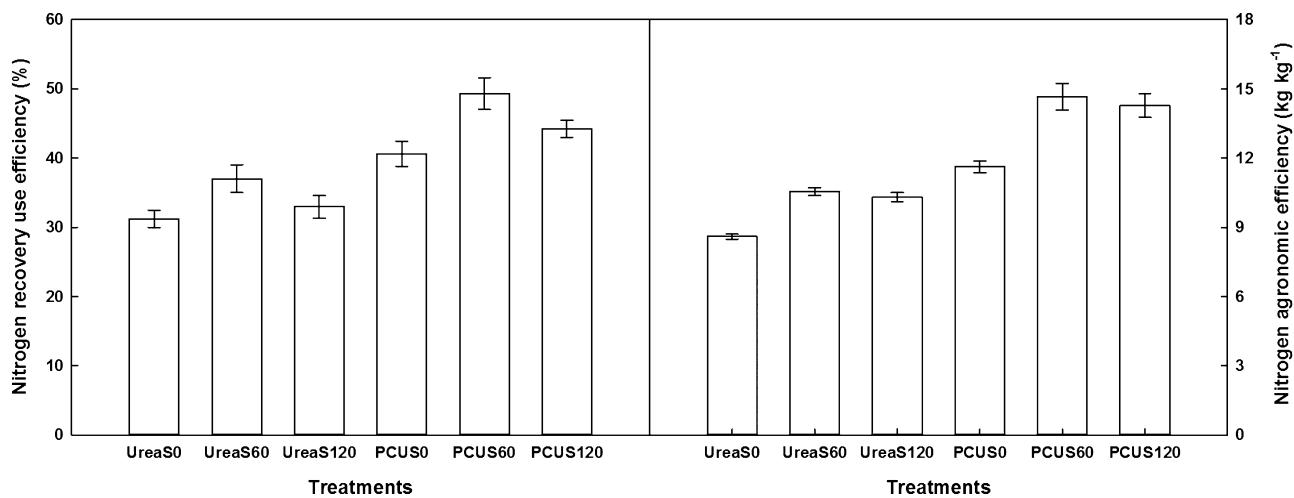


Fig. 5. Nitrogen use efficiency as affected by N and S fertilization. Error bars represent standard error from mean ($n=3$).

3. Results

3.1. Nitrogen cumulative release characteristics of PCU in water and field conditions

The N release rate of PCU was slow in the first month after immersed in 25 °C water, followed by an accelerated release stage (40th to 80th day), and ending with a reduced N-release stage (Fig. 1). The cumulative release rate of PCU reached 89.74% in 120 days. Under field condition, only 14.08% of N was released in the first 30 days, and then the release rate was accelerated from 60th to 120th after buried in the soil. Approximately 84.7% of N had been released at the maturing stage.

3.2. Content of NO_3^- -N, NH_4^+ -N and available S in soil

The contents of NO_3^- -N and NH_4^+ -N were significantly affected by fertilization, and the control (CK) was the lowest in each growth period and showed a decreased trend (Fig. 2). For the urea treatment, the contents of NO_3^- -N and NH_4^+ -N were higher in the

seedling stage, compared with PCU treatments. Nonetheless, it decreased rapidly with the growth and lower than that in PCU treatments after the first bloom stage. Conversely, PCU showed a steady N supply during the whole growth periods, the content of NO_3^- -N was the highest in full boll setting stage, while the NH_4^+ -N was occurred in the initial boll-opening stage. The S fertilizer showed no significant effects on NH_4^+ -N, but the NO_3^- -N content in UreaS60 was higher than that in UreaS0 and UreaS120. The available S content was increased from seedling stage to full boll setting stage, then decreased in the initial boll-opening stage, and ended with an increasing trend in maturing stage (Fig. 3). For each type of N fertilizer, the available S concentration increased as the input S rate increased and the lowest S content was in CK during the whole ontogenetic stages.

3.3. Effects of PCU and S fertilization rates on SPAD values and Pn rate

The SPAD values were considerably affected by N and S fertilization, and also by their interaction, except that in squaring and full

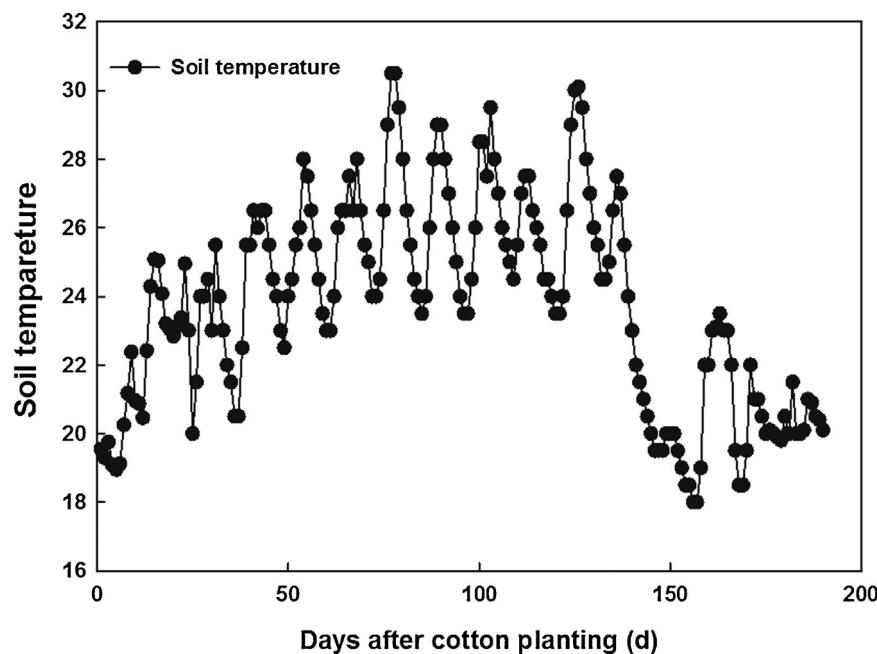


Fig. 6. Constant changes of soil temperature after fertilization.

boll-opening stages (Table 1). Under PCU fertilization treatments, the SPAD values were higher compared with urea treatments, and PCU × S60 treatment achieved the highest SPAD values in each ontogenetic stage, but there was no significant difference among PCU × S60, PCU × S120 and Urea × S60 treatments. The application of 60 kg ha⁻¹ S fertilizer increased the SPAD values by 4.21–17.98% compared with no S fertilization. However, it showed no difference between S0 and S120 treatment, except that in full boll-opening stage. The Pn rate showed an increase trend with the increase of N application, and augmented with the process of growth, but it was reduced from the initial boll-opening stage to maturing (Fig. 4). The Pn rate was lower in PCU treatments in comparison with urea treatments with the same S fertilization rate, and the lowest Pn rate was found in PCUS0. However, the opposite trend was observed from the first bloom stage to maturing stage.

3.4. Effects of PCU and S fertilization rates on the enzymes activities

The activities of NR, GS, SOD and content of MDA were considerably affected by N and S fertilization, but only the activity of NR and MDA contents were significantly affected by their interaction (Table 2). The POD activity was affected by neither S rates; nor N and S interaction. There were significant differences between the N-fertilized treatments, where the PCU treatments were higher than the Urea treatments in enzyme activities. PCU × S60 treatment achieved the highest NR and POD activities. In general, S60 and S120 showed no marked difference, but they were all higher than S0 treatments. Meanwhile, all N-fertilized treatments produced significantly lower MDA contents than the unfertilized control.

3.5. Cotton yield, fiber quality and N use efficiency

The number of bolls, seed cotton yields and lint yields were all affected by the type of N fertilizers and S fertilization rates and their interaction (Table 3). However, the lint percentage was affected neither by the type of N fertilizers and S rates (except that in 2014), nor by their interaction, it maintained between 42.7% and 44.7% among the treatments. The single boll weight showed no significant

difference among all the treatments, except that CK was lower than others in 2015. All PCU-fertilized treatments produced significantly more cotton yield and boll weight in comparison with urea treatments. The PCU treatments achieved significantly higher lint yield by 5.54–11.17% compared to urea treatments, generally, PCU × S60 treatment was the highest than other treatments in 2014, but there was no significant difference among PCU × S0, PCU × S60 and PCU × S120. The lint yields were significantly increased 6.74–9.26% and 3.77–7.46%, respectively, by application of 60 and 120 kg ha⁻¹ S fertilizer than no S fertilization.

As compared to control, the fiber quality was marked improved by N and S fertilization (Table 4). There were obvious differences among the N-fertilized treatments, where the PCU treatments were higher than the urea treatments in fiber length, fiber uniformity and fiber strength. Moreover, fiber length and uniformity was affected by the interaction of N and S. The fiber length and strength in S60 and S120 was increased compared with S0. No difference was observed about micronaire and fiber elongation among treatments.

In general, nitrogen recovery use efficiency (RUE) of PCU treatments was higher in comparison with urea treatments, and also exhibited an increasing trend with increased S fertilization rates under the same type of N fertilization (Fig. 5). The greatest RUE was produced in PCUS60, which was 33.21–57.86% higher than Urea treatments, and also increased by 21.36% and 11.51%, respectively, compared with PCUS0 and PCUS120. Similar trends were observed for AUE, and the highest AUE was occurred in PCUS60.

4. Discussion

4.1. Effects of PCU and S fertilization rates on cotton yield and fiber quality

The cotton plants need nutrients continuously for the growth, but they absorb nutrients differently in quantity and speed, because plants absorb critical and urgent elements in different ontogenetic stages (Xue et al., 2008). Nitrogen is an essential macronutrient that is required most for cotton production (Hou et al., 2007), but N deficiency and poor N use efficiency adversely affect cotton growth and yield (Kawakami et al., 2012). In the present study, the release

rate of PCU was slowly in the first month, and then accelerated, finally dropped under the field condition, the release peak was occurred in the first bloom stage to full boll setting stage. Cotton plants were small in seedling stage and absorbed few N, while the cotton biomass accumulated and N uptake was the fastest from the first bloom to peak bloom stage (Yang et al., 2012a), and consequently, the successive releases of N from PCU corresponded well to the requirements of N in the growth stages of cotton. Nutrient release rates of PCU are known to be significantly affected by temperature and moisture content (Geng et al., 2015). The average soil temperature was 23.77 °C (Fig. 6) after the fertilization, which was lower than that in the laboratory (25 °C water). Thus, the N release longevity of PCU in field condition was longer compared with that in 25 °C water. Conversely, the rapid hydrolyses process of urea caused heavy N losses (Khan et al., 2015), resulted in a lower NO₃⁻-N and NH₄⁺-N concentration after the first bloom stage compared with PCU treatments. Lint yields and NUE were significantly affected by the types of N, S rates and their interaction. PCU and S increased lint yields by 5.54–11.17% and 3.77–9.26%, respectively, compared with Urea and SO. In summary, the effect of sulfur addition had relevance when N was sufficient, showing a positive interaction between PCU and S fertilizer on cotton growth, reflected in a higher NUE, and the similar results were also reported in wheat (Salvagiotti and Miralles, 2008).

Fiber properties are always affected by the interactive effects of genotype, cultivar, weather, and soil conditions (Pettigrew, 2003; Dong et al., 2006; Zeng et al., 2011). For example, Read et al. (2006) supported evidence that N stress indirectly affects cotton growth, as N deficiency decreased fiber length, strength and micronaire, and the correlation between lint yield and fiber strength was affected by genotypes (Zeng and Pettigrew, 2015). In the current study, micronaire and fiber elongation were neither affected by N, nor by S fertilization, which may be the intrinsic quality of the gene. Fiber length, uniformity and strength were improved in PCU treatments compared with Urea treatments, due to the continuous and adequate supply of N from PCU during the whole ontogenetic stages. Fiber length and strength increased with the increased S fertilization rates, but a significant S by N interaction was only observed on fiber length and uniformity. In summary, the consequence of the N × S interaction was evidenced by the higher NUE and fiber qualities when cotton was grown with 60 kg ha⁻¹ S. Therefore, the application of PCU with 60 kg ha⁻¹ S will be of relevance for a better management of the agronomical input in a silt loam soil, where S was deficient in the north China plain.

4.2. Effects of PCU and S fertilization rates on physiological characteristics of cotton leaves

Premature senescence in cotton has been occurring with an increasing frequency in many cotton-growing countries (Dong et al., 2005; Dong et al., 2006). Since leaf photosynthesis is closely correlated with leaf senescence, the Pn of the youngest fully expanded leaf on the main stem has been considered as a valid indicator of plant senescence (Dong et al., 2012). In this study, Pn rates was increased by PCU fertilization from the first bloom stage, and Pn showed a positive correlation with SPAD values, similar effects had also been reported by Buscaglia and Varco (2006). A significant S and N interaction was observed on SPAD values in the first bloom, full boll setting and initial boll-opening stage. Meanwhile, the activity of NR in PCU was extremely higher compared with Urea, due to the NO₃⁻-N was the main N source for this enzyme, and PCU increased the content of NO₃⁻-N. Sulfur addition also increased NR and GS, since S was an essential constituent of enzymes involved in N metabolism (Swamy et al., 2005), its sufficient could lead to an increase in N assimilation.

Reactive oxygen species (ROS) can be continually produced as natural products accompanied by N metabolic processes in plants, and excessive reactive oxygen species will degrade polyunsaturated lipids, forming MDA (Mittler, 2002), POD and SOD are considered to be the major antioxidant enzyme for scavenging ROS. Thus, MDA together with Chl content and Pn have been used as valid indicators of leaf senescence (Zhang et al., 2012a). Generally, the premature senescence frequently developed during the period of rapid boll filling, and resulted in reduced lint yield and poor fiber properties (Dong et al., 2008). The current results revealed that the use of PCU and S improved the antioxidant activity of plant, and reduced the content of MDA, which was a reflection of the degree of membrane lipid peroxidation. Therefore, the application of PCU and S delayed leaf senescence as evidenced by the improvement of Pn, NR, GS, SOD and POD.

5. Conclusions

In the current study, the release rate curve pattern of PCU synchronized with N requirements of cotton. Lint yield and fiber qualities were significantly affected by the types of N, S fertilization, and their interaction. The maximum lint yield and RUE were achieved with PCU and 60 kg ha⁻¹ S fertilizer, which was 5.54–11.17% and 57.86% higher in comparison with urea treatments and SO. The contents of soil NO₃⁻-N and NH₄⁺-N were significantly increased from the first bloom stage to the initial boll-opening stage due to the continuous supply of N in PCU. Fiber length, uniformity and strength pronouncedly promoted, SPAD values and Pn rates increased and enzymes activities enhanced by the application of PCU. In conclusion, PCU combined with 60 kg ha⁻¹ S fertilizers is recommended for gaining greater yields and higher NUE of cotton in the North China plain.

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