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Preparation and Properties of Kaolin Based Slow-Release Fertilizer

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ABSTRACT

A slow-release fertilizer is important to improve soil conditions and enhances food security through continuous plant supply. With urea as a nitrogen source, kaolin as a carrier, acrylic acid as a reaction monomer, potassium persulfate as an initiator, N, N '-methylene bisacrylamide as a crosslinking agent, under the action of cyclohexane and sorbitan monostearate, kaolin based slow-release fertilizer was synthesized by reverse suspension method for the first time. The optimum conditions for the synthesized polymer were 0.6 g kaolin, 2 g urea and 80 ℃ temperature. The maximum water absorption capacity of the slow-release fertilizer was as high as 219.75 g/g. The water retention performance lasted for at least 10 days, and the slow-release property lasted for at least 28 days. The release of nitrogen content was slow and stable. The FTIR analysis indicated that the cross-linking occurred between kaolin, urea, and resin meanwhile, the SEM analysis supported that the product had a special network structure with a rough and porous surface. TGA analysis confirmed the stability of the kaolin-based slow-release fertilizer. The findings fully validated that the synthesis method of slow-release fertilizer was feasible and has the potential to be a popular commercialized product be popularized in areas lacking water or fertilizer.

1. Introduction

Drought, water shortage, a low utilization efficiency of water and fertilizer directly affect food security and the sustainable development of agriculture. In 2018, the effective water utilization coefficient for agricultural irrigation in China is only 0.548, and the nitrogen fertilizer is only about 35%. Such values are far lower than the average level of developed countries [1]. Therefore, solving the demand for chemical fertilizers in agricultural production has become an increasing concern and important issue. Compared with ordinary fertilizers, slow-release fertilizers have the advantages of fast nutrient-controlled release, long fertilizer life cycle, high fertilizer utilization rate, and reduced nutrient diversion loss [2]. Therefore, the use of slow-release fertilizers is an effective method to reduce nutrient loss and environmental hazards [3-4]. However, most of the current water-retaining

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slow-release fertilizers have a high cost, complex process, poor slow-release performance, poor mechanical performance, and poor salt resistance [4].

Urea is a very common fertilizer with an amide structure. It can be used as the main source of nitrogen in slow-release fertilizers. It has excellent hydrophilicity and is easy to lose with water [5-6]. The super absorbent resin is a medium crosslinked hydrophilic material with three-dimensional polymer network structure, which has a strong water absorption capacity [7-8]. It increases the diameter of soil aggregates, improves the permeability of the soil, reduces the biological tensile strength and capacity of the soil, prevents soil hardening, enhances the retention of roots on soil, and greatly reduces water and soil loss [9]. The super absorbent resin can wrap urea and improve the utilization rate of urea fertilizer, but its salt tolerance in soil is poor [10]. Inorganic clay kaolin can be broken by itself in the soil, which not only does not pollute the soil but also improves the soil structure [11]. Kaolin which is also known as ceramic clay effectively improves the absorption of calcium ions by super absorbent resin in the soil, and enhances the salt resistance of resin.

With Kaolin as the carrier and urea as the nitrogen source, the water-retaining slow-release fertilizer was prepared by inverse suspension polymerization for the first time. The optimization synthesis conditions such as kaolin content, urea content, and temperature, were discussed by a single variable method and supported by an orthogonal experiment study (Spss20.0). Besides, water retention performance and nitrogen content released by the synthesized polymer are also investigated. After that, the optimized product of the slow-release fertilizer was characterized using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and thermogravimetric (TGA) analysis. The chemical and physical properties of the slow-release fertilizer were evaluated.

2. Materials and Methods

2.1 Materials

Acrylic acid ($C_3H_4O_2$, MW: 72.06 g/mol, AR), urea (CH_4N_2O, MW: 60.06 g/mol, AR), potassium hydroxide (KOH, MW: 56.11 g/mol, AR), potassium persulfate ($K_2S_2O_8$, MW: 270.32 g/mol, AR), N, N '- methylenebisacrylamide ($C_7H_{10}N_2O_2$, MW: 154.17 g/mol, AR), sorbitan monostearate ($C_{24}H_{46}O_6$, MW: 430.62g/mol, AR), and cyclohexane (C_6H_{12}, MW: 84.16 g/mol, AR) were acquired from Damao Chemical Reagent, Tianjin, China Factory. Kaolin ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$, MW: 258.00 g/mol, AR) was purchased from Tianjin Guangfu Technology Development Co., Ltd.

2.2 Synthesis of Kaolin Based Slow-Release Fertilizer by Inverse Suspension Polymerization

A 9.7 g KOH was dissolved in 30 mL deionized water to prepare a potassium hydroxide solution. The acrylic acid solution was prepared by dropping all the prepared Potassium hydroxide solution into 10 g of acrylic acid in an ice bath [12]. The solution to be polymerized was prepared by adding kaolin, urea, potassium persulfate aqueous solution, and N, N '- methylenebisacrylamide solution into the acrylic acid solution under magnetic stirring condition. A 0.25 g of sorbitan monostearate and 50 mL of cyclohexane were added into a three-necked bottle with a constant pressure drip funnel, a condenser, and a stirrer. The sorbitan monostearate was completely dissolved and dispersed in cyclohexane at 60 ℃ water bath. The solution to be polymerized was slowly dropped into a three-necked bottle [13-14] through a constant pressure-dropping funnel within half an hour. Next, the reaction temperature was controlled at 60 ℃ for duration of 1.5 hours. After the reaction was completed, the polymer product solution was quickly poured out by adding a small amount of cyclohexane, and the impurities in the polymer were removed by washing and soaking the polymer using industrial alcohol [15-16]. The polymer was dried in an oven for 12 hours and then crushed into powder. Finally, kaolin-based slow-release fertilizer was prepared.

2.3 Determination of Water Absorption Capacity of Kaolin-Based Slow-Release Fertilizer

The prepared slow-release fertilizer powder ranging 0.4 – 0.6 g was put into 500 mL tap water, respectively. After water absorption, the slow-release fertilizer was filtered through a nylon mesh bag and weighed [17, 18]. The weight was recorded as *M*. The measured water absorption, W_a (g/g) was calculated using Eq. (1).

$$Wa = \frac{M-M_0}{M_0} \tag{1}$$

where *M* is the weight of kaolin-based slow-release fertilizer after water absorption; M_0 is the sample weight after filtering. The procedures were repeated for parameters of urea amount 2 – 8 g and temperature 75 – 90 ℃ to determine the optimum conditions.

2.4 Determination of Water Retention Performance of Kaolin-Based Slow-Release Fertilizer

Approximately 0.1 g slow-release fertilizer sample was placed in a nylon mesh bag that was prior fully swelled in tap water for 4 hours. The water on the surface of the slow-release fertilizer that filtered by the nylon bag was wiped off, and the weight of the swelling slow-release fertilizer was recorded as *m*. The water in the slow-release fertilizer was slowly volatilized in the air, and the quality became smaller. The slow-release fertilizer was weighed every day, and the weight was recorded as m_i [19]. The percentage of water retention, W_r (%) was calculated by using Eq. (2).

$$Wr = \frac{m_i}{m} * 100 \tag{2}$$

where *m* is the weight of the swelling slow-release fertilizer; m_i is the sample weight after drying.

2.5 Determination of Nitrogen Content of Kaolin-Based Slow-Release Fertilizer

At room temperature, 1 g of the slow-release fertilizer sample was weighed and placed in a nylon mesh bag, and the nylon mesh bag was placed in 1 L of tap water. The sample was withdrawn daily for a duration of 10 days to determine the nitrogen content of the solution in the beaker [20]. The nitrogen content was measured by Hanon automatic Kjeldahl nitrogen analyser (K9860).

2.6 Orthogonal Experimental Study

The quantitative and statistical analysis of the effect of the amount of kaolin (A), amount of urea (B), and temperature (C) factors were investigated on the water absorption performance through orthogonal experiments designing (Table 1). Three levels were set for each factor as in Section 2.3. An orthogonal table L9 was designed in which a blank column was designated for the error evaluation. Procedures in Section 2.2 and Section 2.3 were repeated to synthesize the polymer and investigate the absorption performance, respectively.

Table 1
Orthogonal Test Design Table（Spss20.0）

Number of experiments	Amount of kaolin (g)	Amount of urea (g)	Temperature (°C)
1	0.4	2.0	75
2	0.5	5.0	75
3	0.6	8.0	75
4	0.4	5.0	80
5	0.5	8.0	80
6	0.6	2.0	80
7	0.4	8.0	90
8	0.5	2.0	90
9	0.6	5.0	90

2.7 Characterization Study

The optimized sample was selected to characterize the chemicals and physical properties. Fourier Transform Infrared (FTIR) (Perkin Elmer, Spectrum Two), Scanning Electron Microscope (SEM) (Tescan Vega3), and thermogravimetric analysis (TGA) (Perkin Elmer, TGA 4000) were used for the investigations.

3. Results and Discussion
3.1 Water Absorption Capacity of Kaolin-Based Slow-Release Fertilizer
3.1.1 Effect of kaolin amount on the water absorption capacity

Kaolin amount is an important parameter as it acts as a carrier in the polymer kaolin-based slow-release fertilizer. The water absorption analysis of the slow-release fertilizer based on different amount of kaolin was shown in Figure 1. The water absorption capacity of kaolin based slow-release fertilizer increased first and then decreased with the amount of kaolin. This was because a new polymer with a network structure was formed with the combination of kaolin and super-absorbent resin [21]. The high kaolin amount increased the complexity of the structure of the kaolin-based slow-release fertilizer [22]. Hence, it becomes a strong water-absorbent material with an improved water absorption capacity. However, if the content of kaolin was too high, the water absorption capacity of the reticular structure of the slow-release fertilizer would be reduced to a certain extent.

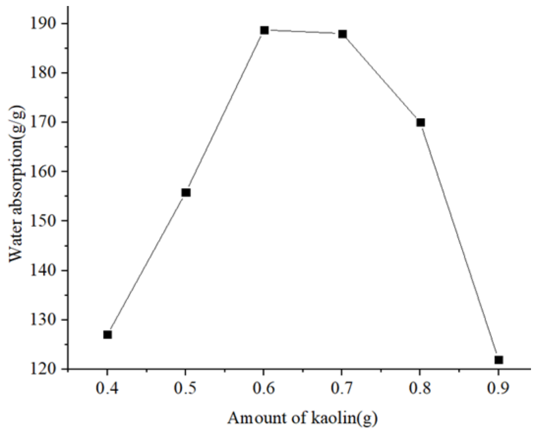

Fig. 1. Effect of kaolin amount on water absorption capacity

3.1.2 Effect of urea amount on the water absorption capacity

Figure 2 illustrates the water absorption capacity of kaolin-based slow-release fertilizer with different amounts of urea. A high amount of urea resulted in a low water adsorption capacity of the kaolin-based slow-release fertilizer. This supported that urea is a low-cost hydrophilic small molecule and has an amide structure to form a super absorbent resin that facilitates the water adsorption of the polymer. However, the excessive amount of urea may impact the water absorption of the kaolin-based slow-release fertilizer due to the interference between ions [12].

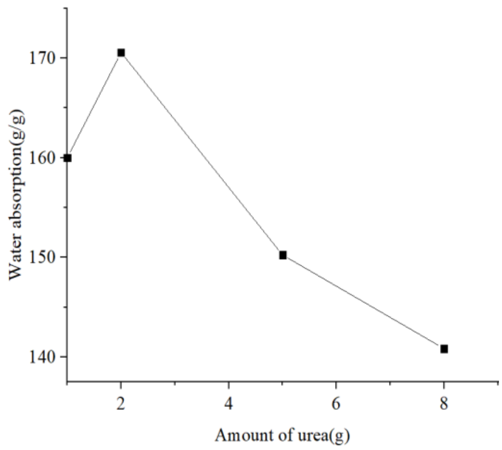

Fig. 2. Effect of urea amount on water absorption capacity

3.1.3 Effect of temperature on the water absorption capacity

The influence of temperature on the water absorption capacity of kaolin-based slow-release fertilizer is shown in Figure 3. When the temperature was 80 ℃, the water absorption capacity of the slow-release fertilizer was the highest, while at 75 ℃ and 90 ℃, the water absorption capacity of the slow-release fertilizer was relatively low. At 80 ℃ optimum condition under the action of the initiator and cross-linking agent, multiple branches and many large porous three-dimensional network polymers were formed.

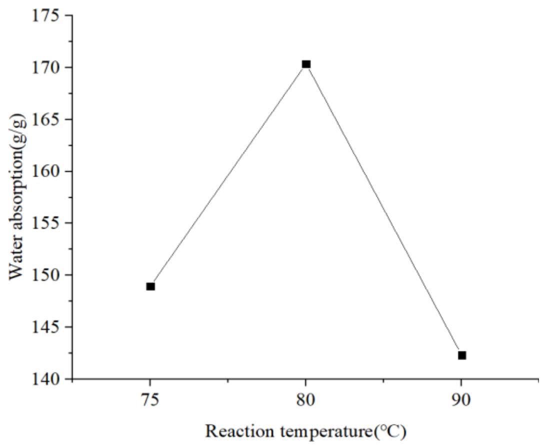

Fig. 3. Effect of temperature on water absorption capacity

Such circumstances increased the water absorption of kaolin-based slow-release fertilizer [23]. When a lower temperature of 75 ℃, the crosslinking agent and initiator were not fully polymerized the mixture, forming short chains, which led to low water absorption capacity [24]. At the high temperature of 90 ℃, too many branched chains were generated under the action of the initiator and crosslinking agent. Moreover, these branched chains were intertwined together [25], hindering the water absorption of kaolin-based slow-release fertilizer.

3.2 Water Retention Performance of Kaolin-Based Slow-Release Fertilizer

Water retention property is an important index to evaluate the performance of kaolin-based slow-release fertilizer. Figure 4 shows the water retention performance of kaolin-based slow-release fertilizer for 10 days at room temperature. A decrease in the water retention performance of the slow-release fertilizer with time was observed. The water retention performance reached about 50% on the fifth day and about 20% on the tenth day. The main reason may due to the super absorbent resin component in the slow-release fertilizer storing a large amount of water, and slowly releasing the water stored in it as the water volatilized. This helped the crops fully absorb the water and effectively improved the utilization rate of water resources [19]. The super absorbent resin also changed the pore distribution of the soil, increased the capillary pore, led to a larger pore proportion, increased the water retention capacity, and improved the water retention performance of the soil in the process of water loss under natural conditions [20]. It concluded that the kaolin-based slow-release fertilizer has good water-holding performance that provides water for continuous crop growth.

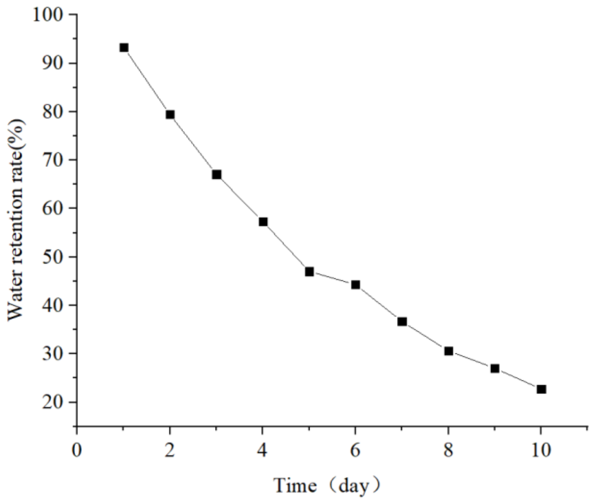

Fig. 4. Water retention performance of fertilizer

3.3 Slow-Release of Nitrogen Content of Kaolin-based Slow-Release Fertilizer

The change in nitrogen content of in solution due to kaolin-based slow-release fertilizer with time is shown in Figure 5. The kaolin-based slow-release fertilizer continuously releases nitrogen with the increase of days, and the nitrogen release curve was approximate a straight linear line. The release of the nitrogen content from the kaolin-based slow-release fertilizer was relatively stable. On the 28th day, the nitrogen content released by the kaolin-based slow-release fertilizer reached 4.042 mg/L. There were two main reasons. One reason may due to the adsorption of urea by the space network structure resin of slow-release fertilizer. In the process of water absorption and expansion of the slow-release fertilizer, the urea in the slow-release fertilizer would slowly release. Another reason may be that during the synthesis of the slow-release fertilizer, part of urea participated in the polymerization reaction and was converted into amide branch chain compounds on the polymer carbon chain [20]. During the water absorption expansion of the slow-release fertilizer, nitrogen was decomposed into movable ions, so that it was slowly released [23]. This indicated that the kaolin-based slow-release fertilizer is an effective long-term fertilizer that provides the required nutrients to the crops continuously to ensure food security [26].

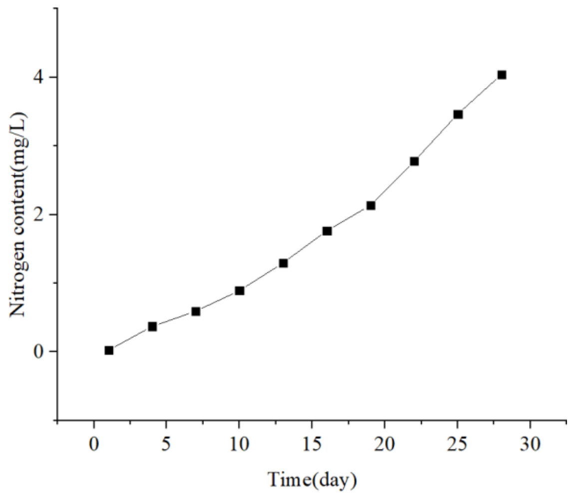

Fig. 5. Nitrogen content change with time

3.4 Orthogonal Experimental Study

Table 2 shows the range and mean values of various factors affecting water absorption that were calculated from the orthogonal test data. The orthogonal design usually is used to test the comparative effectiveness of multiple intervention components to determine the best combination of factor levels. K_1, K_2, and K_3 are the sum of indicators at each level of each factor. K_1 represents the sum of test indicators corresponding to the "1" level. K_2 represents the sum of test index values corresponding to "2" level. K_3 represents the sum of test index values corresponding to "3" level. Line R is called range, which indicates the influence extent of the factor on the result by using the largest K to subtract the smallest K [13]. The greater the range R of each reaction factor [13], the greater the influence of this factor on the water absorption of kaolin based slow-release fertilizer. Through the orthogonal experiment of range analysis, the highest water adsorption capacity was found at 219.75 %. The best synthetic conditions of kaolin-based slow-release fertilizer were 0.6 g kaolin, 0.2 g urea, and 80 °C temperature. The main factor that plays a significant role in water absorption capacity was the amount of kaolin, followed by the amount of urea, and lastly temperature. This finding confirmed the results in Section 3.1 the optimized conditions for the synthesis of kaolin-based slow-release fertilizer.

Table 2
Orthogonal test results

S/N	(A)Amount of kaolin/g	(B) Amount of urea/g	(C)Temperature /℃	(D)Error	Water absorption w_a(g/g)
1	0.4(1)	2.0(1)	75(1)	1	140.52
2	0.5(2)	5.0(2)	75(1)	2	135.10
3	0.6(3)	8.0(3)	75(1)	3	171.22
4	0.4(1)	5.0(2)	80(2)	3	140.34
5	0.5(2)	8.0(3)	80(2)	1	151.05
6	0.6(3)	2.0(1)	80(2)	2	219.75
7	0.4(1)	8.0(3)	90(3)	2	100.24
8	0.5(2)	2.0(1)	90(3)	3	151.41
9	0.6(3)	5.0(2)	90(3)	1	175.28
k_1	*127.03*	170.56	148.95	155.62	
k_2	145.85	150.24	170.38	151.70	
k_3	188.75	140.84	142.31	154.32	
Range R	61.72	29.72	28.07	3.92	
Primary and secondary factors		A>B>C			
Optimal scheme		$A_3B_1C_2$			

3.5 Characterization Study
3.5.1 FTIR analysis

The FTIR was used to analyse the functional groups of kaolin-based slow-release fertilizer, as shown in Figure 6. The wavelength peaks at 2850 cm^{-1} and 2918 cm^{-1} were the characteristic absorption of -C=O and C-N [26]. The wavelength peak at 1165cm^{-1} and 949 cm^{-1} were assigned for -Si-O, and -Al-OH, respectively [27]. These peaks indicated that the preparation of slow-release fertilizer is successful.

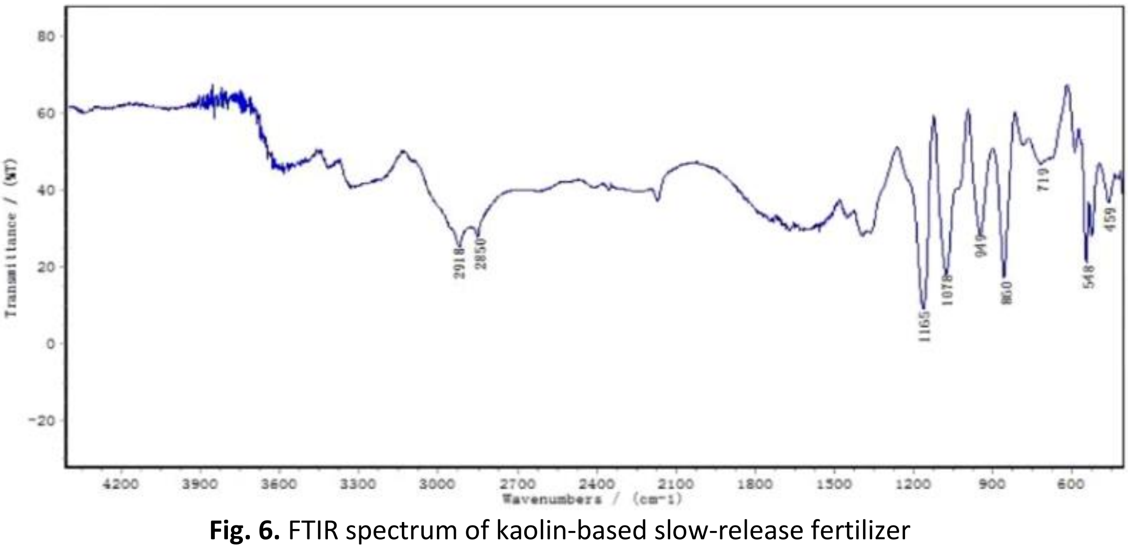

Fig. 6. FTIR spectrum of kaolin-based slow-release fertilizer

3.5.2 SEM analysis

SEM played a very important role in analysing the morphology and microstructure of kaolin-based slow-release fertilizer. The scanning electron micrograph of kaolin-based slow-release fertilizer is shown in Figure 7. The surface of kaolin-based slow-release fertilizer was rough and had obvious holes, indicating that it has a good spatial cross-linking structure [13]. Due to the special structure of kaolin, the slow-release fertilizer absorbed water rapidly and finally led to forming a gel. The slow-release fertilizer after absorbing water in gel form had a tight structure, a larger viscosity, and a higher water-holding capacity [27].

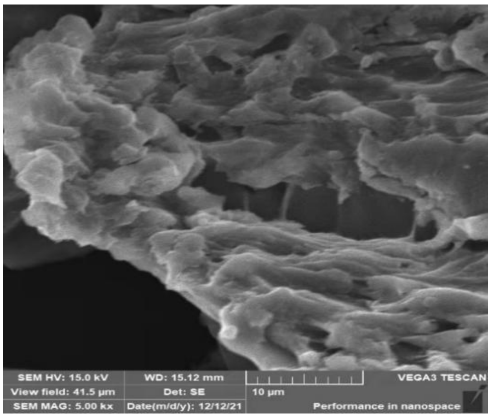

Fig. 7. SEM of kaolin-based slow-release fertilizer

3.5.3 TGA and DTG analysis

Thermo- gravimetry plays a very important role in studying the thermal stability of polymers. Figure 8 and Figure 9 depict the weight of kaolin-based slow-release fertilizer changing with temperature and the relationship between the percentage of weight change of kaolin-based slow-release fertilizer and temperature. The TGA curve of kaolin-based slow-release fertilizer showed the weight of kaolin-based slow-release fertilizer decreased with an increase in temperature (Figure 8). When the temperature was about 480 ℃, the weight of the kaolin-based slow-release fertilizer was basically maintained at about 55% and did not decline further (Figure 8). This indicated that the thermal stability of the kaolin-based slow-release fertilizer was relatively good. According to the DTG curve of kaolin-based slow-release fertilizer in Figure 9, the weight reduction percentage was basically unchanged after 480 ℃. Among them, the slow-release fertilizer at 600 ℃ also contained aluminium and silicon components in the high soil, which were also components contained in the soil itself and would not cause harm to the soil [13,26]. At the same time, the slow-release fertilizer could deposit carbon in the soil and would not cause air pollution [27]. Slow-release fertilizer was an environment-friendly chemical fertilizer.

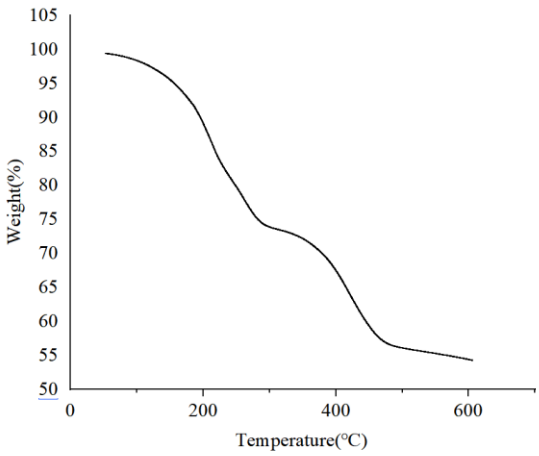

Fig. 8. TGA curve of fertilizer

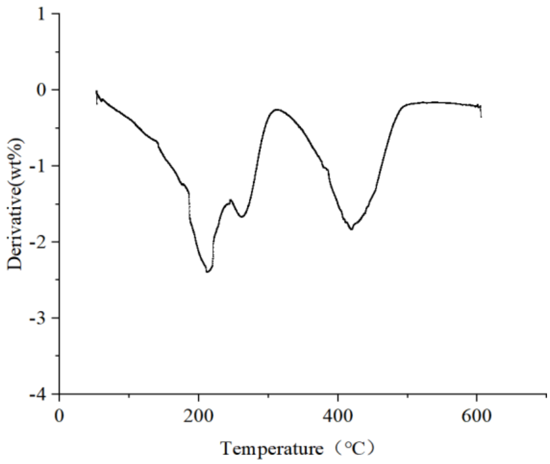

Fig. 9. DTG curve of fertilizer

4. Conclusions

Kaolin-based slow-release fertilizer has been synthesized by inverse suspension polymerization. The synthesis conditions were optimized by orthogonal design and the optimum conditions were kaolin 0.6g, urea 2g, and temperature 80 ℃. Through the determination of water absorption, water retention and nitrogen content of kaolin-based slow-release fertilizer, it was found that the kaolin-based slow-release fertilizer has strong water absorption, strong water holding capacity, and slow nitrogen release. The FTIR, SEM and TGA analysis findings reveal that the polymer has functional groups of C=O、C-N、Si-O、Al-OH, rough surface, porous structure, and stable thermal properties. This kaolin-based slow-release fertilizer has met the requirements of slow-release fertilizer, and supports continuous plant growth for food security and soil amendment purposes.

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