UV polymerization and property analysis of maleacylated methyl cellulose acrylic acid absorbent resin

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In this paper, maleic anhydride (MA) was grafted onto methyl cellulose (MC) and then reacted with acrylic acid to synthesize a high gel strength and fast water absorption resin (AA-co-MC-g-MA) by UV polymerization. The reaction conditions of maleylated methylcellulose (MC-g-MA) were investigated, including the ratio of MC to MA, reaction time and catalyst amount. In addition, the reaction conditions for the synthesis of super absorbent resin were as follows: the amount of MC-g-MA, the degree of substitution of MC-g-MA, polymerization time, and the amount of initiator. Under optimal conditions, the maximum water absorption volume of synthetic resin was 2116 g/g, and the maximum salt absorption rate was 139 g/g. The water absorption resin prepared this time had high water absorption, water retention, excellent pH sensitivity, etc. It was hoped that it will have a good application prospect in the field of industrial production and agriculture in the future.

Keywords: ultraviolet, maleylated methylcellulose, absorbent resin.

INTRODUCTION

China is rich in fresh water resources, but the per capita amount of fresh water resources is small, accounting for only 1/4 of the world per capita¹, and the distribution of China's fresh water resources is very uneven in the region². Located in northwest China, Xinjiang is China's largest province in terms of land area, and its water resources are even scarce. It is not only for human needs, but also for agriculture³. Water retention agent in agricultural production can be used for sowing, seedling and transplanting, soil improvement, ground cover, etc. It is found that it can reduce the death rate of plants, accelerate the growth of crops, enhance soil fertility and reduce the loss of irrigation water. Water retention agent can slow down the release rate of fertilizer and pesticide, and increase the utilization rate and effectiveness of fertilizer and pesticide⁴.

Absorbent resin as a polymer material with special functions was introduced in the United States in 1961. Super absorbent resin is a kind of polymer material with three-dimensional network structure^{5, 6}, which is composed of hydrophilic groups such as hydroxyl, carboxyl^{7, 8}, amide, sulfonic acid and organic carbon chain, and can absorb hundreds or even thousands of times of its own mass of water^{9, 10}. It was widely used in environment, agriculture and forestry, medicine, petrochemical, waste water treatment, communication cable, coal dew treatment, aircraft fuel treatment, food, construction and personal care and other fields¹¹⁻¹⁶.

Super absorbent resin can be roughly divided into the following types: starch resin, fiber resin, synthetic resin and other natural polymer¹⁷. Among them, methylcellulose is a very abundant polymer material and a water-absorbing material that is often used^{18, 19}. Therefore, it has great potential to be synthesized into an environment-friendly high-water-absorbing material by virtue of its characteristics of renewable, low price and environmental friendliness, which can reduce costs and recycle wastes and become an environment-friendly high-water-absorbing material. Maleic anhydride contains unsaturated double bond and acid anhydride structure, which can occur acylation, amidation, decarboxylation, polymerization and other reactions²⁰. The use of maleic anhydride to modify cellulose²¹ and its derivatives and its application to the preparation of superabsorbent resins can introduce hydrophilic groups such as carbon-carbon double bonds and carboxyl groups into the macromolecular chain of superabsorbent resins, which can improved the crosslink density of the resin and the initial water absorption rate. In addition, UV polymerization is a simple, low-cost, short reaction time, clean and efficient initiation method that has developed rapidly in recent years. It has some advantages that cannot be compared with traditional thermal initiation or redox initiation systems. More importantly, it has the advantage of low cost. Therefore, this research group uses this method to synthesize a kind of super absorbent resin²².

Recent studies on super absorbent resins related to acrylic acid are reported as follows: Dongdong Cheng et al.²³ synthesized acrylic acid, urea, potassium persulfate and acrylamide through chemical synthesis to make a super absorbent resin that can absorb water up to 909 g/g. Hisham A. Essawy et al.²⁴ prepared high-absorbent hydrogel by graft polymerization of chitosa-cellulose hybrid acrylic acid, and the water absorption of the obtained structure in distilled water and normal saline was 390 g/g and 39.5 g/g (0.9 wt%), respectively. Qi Shi et al.²⁵ used calcium carbonate modified polymerization to produce (acrylic ester/sodium acrylate) water absorbent resin, the water absorbent of the modified resin is 676.6 times of the dry weight. The lignin supramolecular crosslinked polyacrylic acid absorbent resin with high water absorption (2137 g/g) was prepared by Haonan Zhang et al.²⁶. Shuojun Qu²⁷ synthesized maleic anhydride-acrylic acid salt resistant super absorbent resin in deionized water and 0.9% normal saline water absorption rate of 1476 g/g and 126 g/g, respectively. The absorbent capacity of maleylated cellulose-g-poly(acrylic acid) high-performance absorbent resin synthesized by Amatjan Sawut et al.²⁸ was 1125 g/g in distilled water and 126 g/g in 0.9 wt% NaCl solution.

In this study, the high yield cotton stalk in Xinjiang was used as raw material to extract cellulose, which was then methylated and combined with maleic anhydride, and then grafted with acrylic acid. In the experiment, super absorbent resin was synthesized by UV light polymerization with clean, high efficiency, low cost and short reaction time^{28, 29}. The optimum conditions of synthesis were determined by single factor, the water absorption rate and gel strength of the synthetic resin were studied, and the structure and properties of the super absorbent resin were tested.

EXPERIMENTAL

Materials and instruments

Cotton stalks were from Kashgar, Xinjiang, China. Acrylic acid (analytical pure) was supplied by Tianjin Da MAO chemical reagent factory, China. Sodium hydroxide (analytical pure) was produced by Tianjin Hongyan chemical reagent factory. Sodium chloride (analytical pure) came from Tianjin benchmark chemical reagent co., LTD. Ammonium persulfate and pyridine (analytical purity) was provided by Tianxin fine chemical development center, Tianjin, China. Maleic anhydride (analytical pure) was purchased from Tianjin Shengao chemical reagent factory in China. Acetone (analytical pure) was provided by Tianxin reagent co., LTD.

Self-designed Ultraviolet Light Device (Patent no. ZL201510549926.0) (Fig. 1). The pressurized device used in the test was also made by our laboratory. Fourier transform infrared spectrometer (EQUINOX-55) was purchased from Bruker, Germany. The thermogravimetric analyzer (netzschsta-449c) was also provided by Germany. Scanning electron microscope (LEO1450VP) was from reo, Germany. The frozen drying oven is made in Yuyao, Zhejiang province, China.



Figure 1. Diagram of ultraviolet device

Preparation of MC³⁰

Waste cotton stalks were crushed through 40-mesh sieve, and cellulose was extracted by alkaline hydrogen peroxide method at room temperature. Then 1.0 g of extracted cellulose was soaked in 20 ml 50% sodium hydroxide solution at room temperature for 1h, and the excess sodium hydroxide solution was filtered out. Acetone was used as a solvent, and dimethyl sulfate was added to it drop by drop. The reaction was carried out at 50°C for 1h, and then acetone and dimethyl sulfate were added in the same proportion for 5 cycles successi-

vely. The final product was adjusted to neutral with 10% acetic acid, washed with acetone and dried for later use.

Synthesis of MC grafted with MA

Firstly, an appropriate amount of treated MC was weighed, which was poured into a three-mouth bottle and dissolved in 40 mL water. Secondly, appropriate amount of MA was dissolved in 60 mL acetone and poured into the same three-mouth bottle. Under magnetic stirring, MC and MA were continuously stirred to mix evenly. Then the reaction took place in an oil bath of 60°C for several hours with a few drops of pyridine added as catalyst. At the end of the reaction, the products were washed with acetone and distilled water for several times and centrifuged. After centrifugation, the products were dried in a vacuum drying box and broken into powder for later use.

Synthesis of AA-co-MC-g-MA

Appropriate amount of MC-g-MA was weighed and fully swelled in 10 mL distilled water. A certain amount of ammonium persulfate was dissolved in 2 mL distilled water in a small beaker and poured into a petri dish that was swollen with MC-g-MA.A small beaker was washed with 2 mL distilled water, 5 mL acrylic acid was added, and the neutralization degree was adjusted with 5M NaOH solution. All the reactants in the petri dish were mixed evenly and placed under a 1000 W ultraviolet lamp at temperature of 8–12°C. Then a magnetic stirrer was used to stir the reactants for a certain amount of time. The AA-co-MC-g-MA resin needed for the test was obtained.

Post treatment of resin

The AA-co-MC-g-MA was swollen in 5% acetoneaqueous solution, and the acetone concentration was gradually increased until the white agglomerate appeared³¹. The white precipitate was filtered and dried, and set aside.

Analysis

¹H-NMR was used to determine the chemical structure of resins. An appropriate amount of the dried resin powder was sieved with a 100 mesh sieve, fully expanded in a nuclear magnetic tube containing D₂O, and spectra were obtained in an NMR instrument (VARIANINOVA-400). The FTIR of the resin was measured on the BRUKER EQUINOX-55 infrared spectrometer. Before the test, AA-co-MC-g-MA was pressed into slices with KBr, and the spectrum range was 4000–500 cm⁻¹. Hitachi STA7300 TGA was used to measure the temperature range of 30-800°C, nitrogen atmosphere, nitrogen flow of 30 mL/ min and heating rate of 5°C /min. The appearance of the resin was determined by electron microscope scanner (JSM-5600LV), and the acceleration voltage was 20 kV. Before the test, after AA-co-MC-g-MA was fully swollen in distilled water, freezing it in liquid nitrogen, and then vacuum drying on the freeze dryer for 48 h.

Measurement of water absorption^{28, 32}

Tests conducted in an experimental environment with a relative humidity of 25%: 0.1 g resin particles (through 40–60 mesh sieve) were soaked in 600 mL distilled water or 0.9% NaCl solution to measure its swelling equilibrium and water absorption rate. Measure the water absorption of the resin at different times within 3 h to determine the time to reach the swelling equilibrium. Within 1–30 min., the average suction rate in different time periods was measured at a time interval of 5 min.. The absorbent resin was filtered with 100 purpose gauze bags (weighed after wetting) and hung for 15 min. before weighing to remove excess moisture. The swelling equilibrium (Q) and the absorption rate (v) within a certain time were calculated according to the following two formulas:

$$Q = \frac{m_2 - m_1}{m_1}$$
(1)

$$\upsilon = \frac{Q}{5} \tag{2}$$

Where, m_1 and m_2 are the mass of resin before and after absorption, respectively. Q is the amount of water absorbed per gram of resin sample. v is the amount of water absorbed per gram of resin per minute.

Determination of absorbency of resin under pressure³³

Tests conducted in an experimental environment with a relative humidity of 25%: firstly, the porous glass filter plate (d = 80 mm, h = 12 mm) was placed in the petri dish (d = 160 mm, h = 22 mm). The bottom of the glass cylinder was closed with nylon gauze (d = 60 mm, h = 50 mm, weighed) and placed on the porous glass filter plate. Secondly, the 0.1 g sample is evenly placed on the surface of nylon gauze. The sample was loaded with plastic tank (d = 56 mm, h = 70 mm) which could slide up and down freely in the glass cylinder. The required loads (weighed of different weights) were placed in the tank. Finally, distilled water or salt water was poured into the height of the porous glass filter plate and weighed at different intervals. As can be seen in Fig. 2, the suction volume (AUL) under pressure, the suction rate (ARUL) and the approximate pressure P of the weight per unit area were calculated according to the following three formulas:

$$AUL = \frac{m_2 - m_1}{m_1}$$
(3)

$$ARUL = \frac{m_2 - m_1}{5m_1}$$
(4)

$$P = \frac{\mathrm{mg}}{S} \tag{5}$$

Where m_1 , m_2 and m are the mass before and after the resin suction and the weight, 5 min is the suction



Figure 2. A device for measuring liquid absorption under pressurized conditions

time, g is the gravity acceleration, and its value is 9.8 kg/m², S is the contact area between the weight and the cylinder plastic pipe, and the unit is m^{-2} .

Determination of pH sensitivity of AA-co-MC-g-MA

Solutions with different pH values were prepared by sodium hydroxide and hydrochloric acid. A series of AA-co-MC-g-MA with the same weight and the same quality were soaked in 600 mL solutions with different pH values, and the liquid absorption rate was measured after 5 min. The calculation method of suction rate was the same as formula (1).

Comparison with domestic and foreign resin for diaper

The AA-co-MC-g-MA under the optimal conditions was fully expanded with 5% acetone-water mixture. Then acetone was added to the soluble part to dissolve completely and form white aggregates. It was dried in a drying oven for 48 h, crushed and screened, and compared with the uric diapers used by the American diaper Pampers and used by the domestic diaper Baken (the uric diaper was removed before the experiment and the resin inside was taken out). The water absorption rate and gel strength were measured and compared simultaneously.

RESULTS AND DISCUSSION

¹H-NMR Analysis of MC-g-MA

Compared with Fig. 3 (a) and (b), proton peaks corresponding to each other appeared in (c). Proton peak 3.21–4.94 ppm shown in spectral diagram (c) corresponded to protons of C2, C3, C4, C5 and C6. Proton peak 5.77 ppm corresponded to the proton of C1 in the pyranose ring. These peaks indicated the presence of



Figure 3. (a), (b) and (c) are the 1H NMR spectra of maleic anhydride, methyl cellulose and modified cellulose respectively

IR Analysis of AA-co-MC-g-MA

Fig. 4(a) described that the characteristic absorption peaks of MC at 1056 cm⁻¹ and 1158 cm⁻¹ in the infrared diagram of (MC) appeared in the infrared spectrum of (MC-g-MA) graft copolymer. Compared with the two spectra, the new characteristic peaks appeared in the spectra of MC-g-MA, namely, 1732 cm⁻¹ (-COOR), 1716 cm⁻¹ (-COOH) and 1635 cm⁻¹ (>C=C<). This indicated that MA was successfully grafted onto MC.

TG analysis of AA-co-MC-g-MA

Thermogravimetric analysis (TG) of AA-co-MC-g-MA resin was shown in Fig. 4 (b). It can be seen from the figure that AA-co-MC-g-MA lost about 3% of its weight below 170°C, which was due to the fact that water does not completely evaporate in the drying process. From 170°C to 190°C, the resin lost about 22.4% of its weight at this stage, which may be due to carboxyl removal on the side chain of the polymer and the break of the methyl cellulose chain. Finally, from 390°C to 500°C, the mass loss of the resin decreased sharply, with a weight loss of about 27.3%, which meant that the cross-linked structure of the polymer was destroyed and the main chain



Figure 4. (a), (b) and (c) are the infrared spectrum of modified methyl cellulos and methyl cellulos, TGA diagram of the resin and SEM diagram of the resin respectively

disintegrated, and the AA-co-MC-g-MA resin basically disintegrated completely^{28, 29, 34, 35}. This was sufficient to show that the prepared resin had good thermal stability at less than 500°C.

SEM Analysis of the AA-co-MC-g-MA

Fig. 4(c) described the external surface morphology of AA-co-MC-g-MA. The picture portrayed that the AA-co-MC-g-MA had a porous, multi-layer structure, which was conducive to the rapid absorption of water. Its structure presented an obvious spatial multi-layer network structure, and the resin skeleton was bonded by hydrogen bonds, resulting in the internal water loss. There were also a number of interrelated irregular sized pores distributing in the gel matrix. This porous microstructure increased the resin's surface area and capillary effect, which led to easier transport of water into the polymer. There were also large open pores in the resin that acted like small reservoirs stored water, making it easier and faster for the liquid to spread through the resin.

Determination of modification conditions of MC-g-MA

The modification conditions of MC-g-MA were determined according to the degree of substitution of hydroxyl group in MC replaced by MA and the water absorption rate of acrylic resin grafted by MC-g-MA. In Table 1, (MC: MA) = 1:3 was determined and 4 h was determined as the modification time. The amount of catalyst was investigated. 4 h was determined as the modification time and 1% was determined as the amount of catalyst. The ratio of (MC: MA) was investigated. The (MC: MA) = 1:3 was determined and 1% was determined as the dosage of catalyst. The modification time was investigated. Combined with Fig. 5(a), when the water absorption rate of AA-co-MC-g-MA resin was at its maximum, the optimal degree of substitution of MC--g-MA was 0.568. The optimal conditions for this result were (MC: MA) =1:3, the modification time was 4 h, and the amount of catalyst was 1% of methyl cellulose.

 Table 1. Determination of modification condition of methylcellulose

	w(MC:MA)	w(Catalyst:MC)/%	Time/h	DS
1	1:1	1.0	4	0.203
2	1:3	1.0	4	0.339
3	2:3	1.0	4	0.329
4	1:3	0.5	4	0.367
5	1:3	1.0	4	0.568
6	1:3	1.5	4	0.602
7	1:3	1.0	3	0.662
8	1:3	1.0	4	0.432
Q	1.3	1 0	5	0.678

Influence of substitution degree of MC-g-MA

The effect of the degree of substitution of MC-g-MA on the water absorption performance of AA-co-MC-g-MA was shown in Fig. 5(a). The substitution degree of modified resin was too low, which led to the low crosslinking density of polymer and the low water absorption rate. Too high degree of substitution produced more crossconnection points on the polymer chain, increasing the degree of cross-linking of the polymer network, resulting in a rigid structure, which made the resin difficult to expand and unable to absorb water quickly. When the



Figure 5. Curves (a), (b), (c) and (d) respectively show the influence of substitution degree of modified methyl cellulose, the influence of modified methyl cellulose, polymerization time and initiator amount on the water absorption rate of the synthesized resin

degree of substitution of Mc-g-MA was 0.568, the AA--co-MC-g-MA had the highest water absorption.

Influence of MC-g-MA dosage

Fig. 5(b) depicted that when the MC-g-MA content was less than 12%, the water absorption rate of resin increased with the increase of MC-g-MA content, but decreased when the MC-g-MA content was more than 12%. The increase in MC-g-MA content meant the increase in the amount of maleyl in the mixed system, which reacted easily with monomers and facilitated crosslinking of polymer chains; when the content of MC-g-MA was more than 12%, some insoluble components increased, and the outer layer of the liquid was difficult to be penetrated by ultraviolet light, leading to its slow or incomplete graft polymerization, which led to the reduction of the absorption rate of resin. Therefore, the MC-g-MA dosage was12 %.

The effect of aggregation time

Fig. 5(c) depicted the effect of polymerization time on the absorption rate of resin, which increased with the increase of exposure time in the previous time period. When the irradiation time was 50 minutes, the absorption rate was the fastest. And then when the exposure time increased, the absorption rate decreased. When the polymerization time increased appropriately, free radicals were produced continuously, which were beneficial to the chain growth reaction and the formation of three-dimensional network structure. However, excessive exposure time led to an increase in the crosslinking density of the synthetic resin, which blocked the pore structure of the resin and slowed down the absorption rate of the resin. Therefore, the optimal polymerization time was 50 min.

Effect of initiator dosage

Fig. 5(d) depicted that when the amount of initiator was less than 0.4%, the water absorption rate of resin increased with the increase of the amount of initiator, and the water absorption rate reached the maximum when the amount of initiator was 0.4%. When the amount of initiator was increased within the range of 0.2% to 0.4%, the chain radical increased continuously, accelerating the monomer conversion rate and formed an appropriate cross-linked network structure, thus accelerating the water absorption rate of resin. When the initiator dosage exceeded 0.4%, more free radicals were produced, which accelerated the chain termination reaction, leading to the formation of homopolymers rather than graft copolymers.

Determination of swelling equilibrium of resin^{28,29}

The swelling equilibrium and water absorption rate curve of AA-co-MC-g-MA were shown in the Fig. 6(a)



Figure 6. (a), (b), (c) and (d) are the swelling equilibrium in distilled water and 0.9 wt%NaCl solution, the water absorption rate in distilled water and 0.9 wt%NaCl solution, the water absorption rate of the resin under pressure and the pH sensitivity of the resin respectively

and (b). The water absorption rate of AA-co-MC-g-MA in distilled water and brine was very fast at first, especially in the first 5 min., and then decreased with the increase of water absorption time. This was because at the beginning of the resin surface had more pore structure, so that the physical adsorption was faster. After absorbing water for a period of time, the pores on the surface of the resin were blocked, and the water absorption rate started to slow down. The water absorption rate of resin in distilled water was higher than that in brine because the osmotic pressure in distilled water was higher than that in brine. Finally, at about 60 min, the water absorption of the resin reached the equilibrium of swelling. As can be seen in the figure, when the resin was in equilibrium, the water absorption in distilled water was 2116 g/g and the water absorption in brine was 139 g/g. In figure (b), the maximum water absorption rate of the resin in distilled water was 157 g/ g.min, and that of brine was 18 g/g.min within the first 5 minutes.

Determination of water absorption of resin under pressure

Fig. 6(c) portrayed that the water absorption rate of the resin in both solutions decreased with the increase of pressure, which was because the load of the ballast resin flattened the pore structure of the resin and the water absorption rate of the resin was hindered. Com-

pared with distilled water, the absorption rate of resin in normal saline was less affected by load, and within the range of 2000–8000 Pa, the absorption rate of resin in saline was narrower. Even if the pressure increased to 8000 Pa, the resin synthesized in this experiment also had a fairly high absorption rate, that is, it could reach 8 g/ g.min in distilled water and 3 g/g.min in 0.9 wt% NaCl solution.

Determination of pH sensitivity of AA-co-MC-g-MA

As can be seen from Fig. 6(d), the AA-co-MC-g-MA had a high liquid absorption rate within the range of pH = 1-13. When the pH of solution was 1, the absorption rate of resin was 25 g/g.min. When the solution pH was 13, the resin absorption rate was 33 g/g.min. However, it was obvious that the solution pH value was too low or too high to affect the resin absorption rate. When the pHvalue of the solution was within the range of 1-7, AAco-MC-g-MA resin existed in relatively acidic conditions, a large number of COO⁻ existed in the form of COOH on the network chain, charged particles were reduced, and the corresponding amount of Na⁺ charge inside the network was also reduced, resulting in a decrease in the osmotic pressure difference between the inside and outside of the resin, and the liquid absorption rate of the resin was also reduced. When the pH value of the

solution was within the range of 7–13, the resin existed in the alkaline environment, the carboxyl groups on the network chain mainly existed as carboxyl ions, and the corresponding number of Na⁺ inside the network chain also increased, so that the osmotic pressure inside and outside the resin was greatly reduced, and the absorption rate of AA-co-MC-g-MA was sharply reduced^{36–38}. The high performance water absorbing resin had strong acid and alkali resistance and was expected to be used as soil water retaining agent in agriculture.

Comparison with domestic and foreign resin for diaper

The water absorption of the synthetic resin was compared with that of the resin used in China's Baken nappies and the America's Pampers nappies, as shown in Table 2. The resin synthesized in the experiment had a higher water absorption rate in distilled water and 0.9 wt% NaCl solution than the resin used in Pampers diapers. As can be seen from the table, the resin synthesized in the experiment had a higher water absorption rate in distilled water and 0.9 wt% NaCl solution than the resin used in Pampers, which was similar to that used in Baken. However, AA-co-MC-g-MA used in this experiment had a wide range of raw materials, the purpose of the experiment was waste utilization, and the use of UV synthesis was simple and clean, water absorption was also comparable to commercial supplies.

CONCLUSIONS

In this work, AA-co-MC-g-MA fast absorbent resin was prepared by etherification, modification of methyl cellulose and grafting of acrylic acid. The optimum synthesis conditions of Mc-g-MA were determined as follows: MC: MA was 1:3, reaction time was 4h, and catalyst amount was 1% of methyl cellulose. In addition, the optimal reaction conditions of synthetic resin were as follows: the degree of substitution of MC-g-MA was 0.568, the dosage of Mc-g-MA was 12% of MC, the polymerization time was 50 min., and the dosage of initiator was 0.4 %of MC. By FTIR and NMR characterization, MC-g-MA and fast absorbent resin were successfully synthesized. SEM showed that the prepared water absorbent resin has multi-void network structure. Moreover, it can be seen from the TGA diagram that AA-co-MC-g-MA has certain thermal stability.

Under the best conditions, the water absorption capacity of water absorption resin after acetone treatment was 2116 g/g, and the salt absorption capacity was 139 g/g. The water absorption rate and salt absorption rate per minute were 157 g/g.min and 18 g/g.min. The synthetic absorbent resin also had good pH sensitivity. Based on the above excellent properties, the fast water-absorbing resin prepared and synthesized in this work will certainly be developed in industry and agriculture.

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Table 2. Comparison of water absorption rate with domestic and foreign diaper resin

	AA-co-MC-g-MA		Pampers		Baken	
	Liquiduptake	Fluid absorption	Liquid	Fluid absorption	Liquid	Fluid absorption
	[g/g]	rate[g/g·min]	uptake[g/g]	rate[g/g · min]	uptake[g/g]	rate[g/g · min]
Distilled water	2116	157	1395	93	2156	160
Physiological saline	139	18	79	10	142	18

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