



Research on Preparation of Flocculants for Sugar Factory Wastewater Treatment

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Authors' contributions

The work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

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ABSTRACT

Polyacrylamide, formaldehyde and carbamide or guanidine carbonate were used as raw materials, cationic polyacrylamide flocculants were synthesized with Mannich reaction, effects of technical conditions on rate of turbidity removal were investigated, and synthesis technics was optimized. Experimental results showed that the flocculation properties of the products prepared with Mannich were better than ones of blank experiments (polyacrylamide). In the same dosage of flocculants, consumption of polyacrylamide in the products was smaller than (or equal to) 1/10 of polyacrylamide (blank experiments), reducing flocculation cost greatly. In the real wastewater treatment of sugar factories with the prepared products under optimum conditions, the turbidity removal rates was approaching to 90% or greater than 90% showing flocculation effectiveness was better. In addition, the wastewater of sugar factories was treated with prepared products and the removal of COD was 10~30%, which showed that using flocculating agents could reach the purposes of wastewater pretreatment.

Keywords: Flocculant; polyacrylamide; carbamide; guanidine carbonate; wastewater.

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1. INTRODUCTION

Sugarcane grows mainly in south and southwest of China, specially in Guangxi, Yunnan, Fujian, and Guangdong. Guangxi province is the largest producer of cane sugar in China. With more than 2.6 million farmers in Guangxi for sugar cane plantation, the planted areas and sugar yields take up over 50% nationwide, and its quantities of cane sugar production and marketing account for 70% through the whole country. Sugar industries play an important role in economic development of country, but effluents released produce a high degree of organic pollution in both aquatic and terrestrial ecosystems and cause health hazards. There are two major wastewater sources: one is from sugar mills, and the other is from the cane molasses distilleries. The sugar factories in Guangxi are the biggest water consumers and effluent generators. With the growth in sugar production, the environmental problems associated with sugar factories are also increased.

The wastewater, from sugar mills and the cane molasses distilleries, contains high concentrations of organic materials, and is high acidity and dark color. Its solid content reaches 10%~12%. Therefore, it is necessary to carry out sugar wastewater flocculation treatment before aerobic treatment, in order to reduce the concentration of COD, the solid content, and the load of aerobic treatment.

In this work, polyacrylamide, formaldehyde and carbamide or guanidine carbonate were used as raw materials, cationic polyacrylamide flocculants [1-5] were synthesized with Mannich reaction. This study had the advantages of simple preparation, high efficient wastewater treatment, low drug consumption and low cost of wastewater treatment.

2. EXPERIMENTAL PROCEDURES

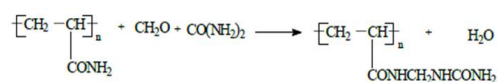
2.1 Materials

Polyacrylamide (300W molecular weight, referred to as PAM (300W)), carbamide (AR), guanidine carbonate (AR) and formaldehyde (40% by weight) (AR) were obtained by a local market.

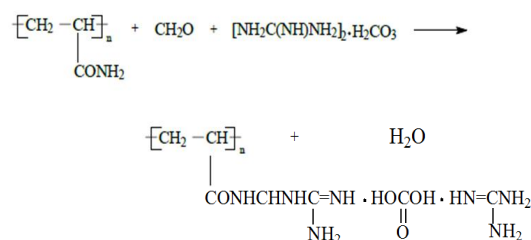
2.2 Synthesis

Polyacrylamide, formaldehyde and carbamide or guanidine carbonate were used as raw materials, cationic polyacrylamide flocculants

were synthesized with Mannich reaction. The over-all reaction is presumed to be



or



2.3 Procedure

To a stirred reactor which provided with a reflux condenser, 60 ml H₂O, 2 g PAM (300W), formaldehyde (40% by weight) and urea or guanidine carbonate was proportionally added. The reaction temperature was kept at 20~60°C for 1 h. Then pH value of the reaction mixture was adjusted to 5.5 with acetic acid, and the reaction continued for 3~6h at this temperature. Finally, pH was adjusted to 7.5 with NaOH, the reaction continued for a certain time and products were obtained. The reaction mixtures were used as flocculants for sugar wastewater flocculation treatment.

2.4 Analytical Methods

The performance of the prepared flocculants is mainly investigated for its ability to remove suspended particles or colloidal particles, namely, the ability to remove turbidity of water. Turbidity of water is one of the important criteria for evaluating water quality. Turbidity was determined by spectrophotography according to GB 13200-91 [6]. The determination of flocculation effectiveness was measured according to Y/T 5796-93 [7].

The rate of turbidity removal was calculated as follows:

The rate of turbidity removal=

$$\frac{\text{turbidity of water sample before treatment} - \text{turbidity of water sample after treatment}}{\text{turbidity of water sample before treatment}} \times 100\%$$

3. RESULTS AND DISCUSSION

Taking the turbidity removal as the object function, and the conditions such as reaction temperature, reaction time, molar ratio of formaldehyde to amine, amine as variable factors, we evaluated the effects of these four factors on the function. Experiments were carried out by using the orthogonal table of $L_{16}(4^4 \times 2^3)$ [8]

[i.e., an orthogonal table of 16 rows (16 times of test), 7 columns (4 factors and 3 empty columns: A= reaction temperature, B= reaction time, C= formaldehyde/ amine, D= amine), 4 levels: 20°C, 30°C, 40°C, 50°C; 2 h, 3 h, 4 h, 5 h; 2.0, 2.2, 2.4, 2.6; 2 levels: 9 g carbamide, 27 g guanidine carbonate], whose levels and factors are shown in Table 1. The orthogonal test results and range analysis are shown in Table 2.

Table 1. Levels and factors in orthogonal test

Levels	A reaction temperature (°C)	B reaction time (h)	C formaldehyde/ amine (mol/mol)	D amine (g)
1	20	2	2.0	9 (carbamide)
2	30	3	2.2	27(guanidine carbonate)
3	40	4	2.4	
4	50	5	2.6	

Table 2. Orthogonal test results and range analysis*

Test number	A	B	Empty column	C	D	Empty column	Empty column	turbidity removal %
1	1	1	1	1	1	1	1	83.18
2	1	2	2	2	1	2	2	77.09
3	1	3	3	3	2	1	2	68.80
4	1	4	4	4	2	2	1	78.00
5	2	1	2	3	2	2	1	76.17
6	2	2	1	4	2	1	2	72.48
7	2	3	4	1	1	2	2	55.18
8	2	4	3	2	1	1	1	69.57
9	3	1	3	4	1	2	2	72.81
10	3	2	4	3	1	1	1	82.48
11	3	3	1	2	2	2	1	72.10
12	3	4	2	1	2	1	2	73.80
13	4	1	4	2	2	1	2	74.97
14	4	2	3	1	2	2	1	54.50
15	4	3	2	4	1	1	1	52.40
16	4	4	1	3	1	2	2	68.67
K_1	307.08	307.14	306.92	266.67	561.39	575.90	587.05	
K_2	273.4	286.55	273.55	293.73	570.83	556.32	545.16	
K_3	301.19	248.48	261.48	296.12				
K_4	250.54	290.04	290.26	275.70				
k_1	76.77	76.79	76.73	66.67	70.17	71.99	73.38	
k_2	68.35	71.64	68.39	73.43	71.35	69.54	68.14	
k_3	75.3	62.12	65.37	74.03				
k_4	62.64	72.51	72.56	68.92				
R	56.54	58.66	45.44	29.45	9.44	19.58	41.90	
Optimum	$A_1B_1C_3D_2$ or $A_1B_1C_3D_1$							

* Dosage of flocculants: 15 mg/L

K_i : Sum of turbidity removal of the level i

k_i : Average of K_i

R (range): $R=k_{max}-k_{min}$

With range analysis of effects of each factor on the rate of turbidity removal, we found that, among these factors, the order of influence upon turbidity removal was as follows: reaction time > reaction temperature > molar ratio of formaldehyde to amine > carbamide or guanidine carbonate.

According to the conclusion of the orthogonal test, the optimum conditions were determined by this test. The optimum conditions are as follows:

Reaction temperature:	20°C;
Reaction time:	2 h;
Molar ratio of formaldehyde to amine:	2.4:1;
Amine:	9 g carbamide or 27 g guanidine carbonate

Through repeated experiments under the optimum conditions, the rate of turbidity removal could reach 90% or greater than 90%, when the dosage of flocculants (solid content) was 10 mg/L, which shows that the synthesized flocculants was high flocculation efficiency.

In the real wastewater treatment of sugar mills with the prepared products under optimum conditions, the experimental results are shown in Fig. 1 (the wastewater from Nanning sugar mill:

dark color; turbid; COD 562.85 mg/L). It indicates that the turbidity removal rates of products are greater than ones of PAM (300W) (blank experiments). When the dosage is 15 mg/L, the turbidity removal rates of blank experiments, products from urea or guanidine carbonate are 77.14%, 94.95% or 92.93% respectively, which shows flocculation effectiveness better. In the same dosage of flocculants, consumption of polyacrylamide in the products was smaller than (or equal to) 1/10 of polyacrylamide (blank experiments), reducing flocculation cost greatly. When the dosage of PAM(300W) (blank experiments) is 10mg/L, the consumption of polyacrylamide(the product from urea) is 1 mg (the reaction mixture (g): 2 g polyacrylamide+60 g H₂O +27 g formaldehyde (solution)+9 g urea =98 g; the ratio of polyacrylamide: 2/98=0.02; solid content of the products: about 20%; the additive amount of the product for flocculation: 10/0.2=50 mg; the consumption of polyacrylamide: 50*0.02=1 mg). Through analysis, it is known that the synthetic products have high efficiency, less consumption of polyacrylamide and low cost of administration, which can save the cost of water treatment.

In addition, the wastewater of sugar mills was treated with prepared products and the removal of COD was 10~30%, which showed that using flocculating agents could reach the purposes of wastewater pretreatment.

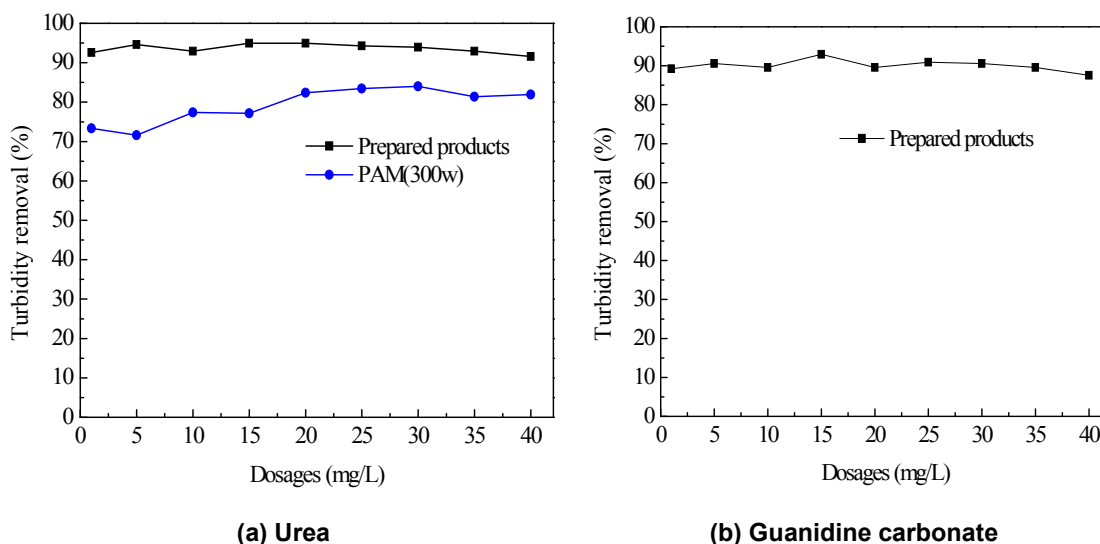


Fig. 1. Real wastewater treatment of sugar factories with prepared products under optimum conditions

4. CONCLUSIONS

1. By orthogonal test main technical parameters of preparing flocculants were optimized. The optimum conditions were as follows: reaction temperature: 20°C; reaction time: 2 h; molar ratio of formaldehyde to amine: 2.4:1; amine: 9 g carbamide or 27 g guanidine carbonate.
2. In the same dosage of flocculants, consumption of polyacrylamide in the products was smaller than (or equal to) 1/10 of polyacrylamide (blank experiments), reducing flocculation cost greatly. In the real wastewater treatment of sugar factories with the prepared products under optimum conditions, the turbidity removal rates were approaching to 90% or greater than 90%, showing flocculation effectiveness better. In addition, the wastewater of sugar factories was treated with prepared products and the removal of COD was 10~30%, which showed that using flocculating agents could reach the purposes of wastewater pretreatment.
3. This study had the advantages of simple preparation, high efficient wastewater treatment, low drug consumption and low cost of wastewater treatment.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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