Study on Optimization of Compound Flocculant's Purification Process of Sugarcane Juice

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Abstract. Sugarcane juice contains a lot of non-sugar organic impurities, and most of the non-sugar organic impurities are suspended in the sugarcane juice in the form of colloidal particles, making the juice turbid. Flocculant is an indispensable clarification aid in the cane juice cleaning process, and its use effect will directly affect the production cost of sugar cane sugar and the quality of white granulated sugar products. This paper is based on the sulfite process of sugar production. After the sulfur smoked juice is heated, the composite flocculant is mixed with the sugarcane juice and then settled. The clear juice is taken to determine the physical and chemical indicators, and the clarification effect of the composite flocculant is investigated. The response surface method is optimized to obtain the composite The best material ratio of the flocculant is PAM 1.70ppm, CTS 1.90ppm, diatomaceous earth 2.10ppm, and the compound flocculant with the best material ratio is used for the cane juice cleaning process experiment. The orthogonal test method optimizes the cane juice cleaning process as The cleaning effect is best when the added amount of phosphoric acid is 160 ul/L, the pre-ash pH value is 6.4, and the neutralization pH value is 7.4, the obtained clear juice is clear and bright, and the color value is low.

1. Introduction
Cane juice cleaning is one of the most critical links in the sugar production process. At present, the clarification aids used in the sulfurous acid method mainly include lime, phosphoric acid, sulfur dioxide, and polyacrylamide (PAM) [1]-[2], among which PAM has a molecular chain Long, there are a large number of chemically active groups that can connect with a large number of precipitated particles to form a floc precipitation, which plays a role of impurity removal and decolorization, but the decomposition products of PAM have carcinogenic effects, and a small amount of residues in white sugar will affect product safety [3]. Chitosan (CTS) is widely used in the food industry as a natural polymer flocculant, safe and non-toxic [4], but its flocculation effect is worse than PAM. Diatomite has strong adsorption performance, light bulk density, uniform fineness, neutral pH, non-toxic, and good mixing uniformity. It is widely used as a filter aid in food, medicine and water treatment [5]-[6], but because of its Adsorbing sucrose molecules takes away the sugar and is
generally not used alone. Improving the clarification efficiency of cane juice, reducing production costs, and reducing the use of PAM are problems that sugar companies need to solve urgently. This paper uses PAM, CTS, and diatomaceous earth as raw materials to prepare a composite flocculant, and studies the best process for cleaning cane juice, providing theoretical data for improving sugar production technology.

2. Test materials and methods

2.1. Test materials and reagents

Raw material: Guangxi LaibinXianggui Sugar Co., Ltd. production line mixed juice (refractive hammer is 17° Bx, PH value is 5.8).

Reagents: 85% phosphoric acid (Xilong Science Co., Ltd.); calcium oxide (Chengdu Kelong Chemical Co., Ltd.); Sulfurous acid (Xilong Science Co., Ltd.); PAM (cation) (Tianjin Damao Chemical Co., Ltd.) Reagent Factory), CTS (Kool Chemical Technology Co., Ltd.), Diatomite (Tianjin Damao Chemical Reagent Factory).

2.2. Test equipment

PH meter (Shanghai INESA Scientific Instrument Co., Ltd.); 722s visible spectrophotometer (Shanghai Qinghua Technology Instrument Co., Ltd.); Abbe refractometer (Shanghai INESA Physical Optical Instrument Co., Ltd.), 1900c portable turbidity meter (Shanghai Shiulu Instrument Co., Ltd.).

2.3. Experiment method

2.3.1. Sample pretreatment. Take 500ml of mixed juice, add lime milk to pre-ash to PH=6.4, heat to 65°C; add 13.3ml sulfurous acid, 0.85% phosphoric acid 8ml, stir evenly, add lime milk to neutralize to PH=7.2, and become the sulfur smoke intensity of 16. Neutralize the juice for later use.

Take 500ml of neutralization juice, heat it to boiling, and divide it into 5 100ml sample liquids, put them into 250ml conical flasks, add quantitative flocculants, stir evenly, let stand for 10min, filter, and take about 50ml of clear juice. Cool to room temperature for later use.

2.3.2. Determination of clear juice index. An electromagnetic PH meter was used to determine the pH value of the clear juice; a turbidimeter was used to determine the turbidity of the clear juice; a refractive hammer meter was used to determine the hamerness of the clear juice; a spectrophotometer was used to determine the absorbance of the clear juice and calculate its color value.

Calculation of color value of clear juice: color value \( IU_{560}=1000A_{560}/bc \) [1]

In the formula, \( IU_{560} \)--the international sugar color value of the sample solution measured with a wavelength of 560nm, IU;

\( A_{560} \)--The absorbance of the sample solution measured with a wavelength of 560nm;

\( b \)--The thickness of the cuvette (cm)

\( c \)--Solid solution concentration of sample solution (g/ml)

\( c=B/d_{20}^{20}/100 \)

In the formula, \( d_{20}^{20} \)--apparent relative density (20°C) (g/ml)

\( B \)--Refractive hammer degree (°Bx)

2.4. Design of test plan

2.4.1. Single factor test. Investigate the influence of PAM, CTS, and diatomaceous earth addition on the cleaning effect of sugarcane juice. The addition of flocculants were 2.0ppm, 2.8ppm, 3.0ppm, 3.2ppm, 4.0ppm, and the color value, hamerness and turbidity of the clear juice were determined, PH value, each test is repeated three times, and the average value is taken to reduce the error.
2.4.2. Response surface optimization test. On the basis of the single factor test, select PAM, CTS, diatomite three flocculants to design a three-factor three-level response surface test, take the clear juice color as the response value, repeat each group of tests three times, and take the average value to reduce the error. Optimize the optimal material ratio of the composite flocculant. The response surface test plan is shown in Table 1.

| Level | PAM (ppm) | CTS (ppm) | Diatomite (ppm) |
|-------|-----------|-----------|-----------------|
| -1    | 0         | 0         | 0               |
| 0     | 1.5       | 1.5       | 1.5             |
| 1     | 3         | 3         | 3               |

2.4.3. Orthogonal design. The compound flocculant with the best material ratio was used to carry out the cane juice cleaning process experiment, and the three main factors affecting the cleaning effect of cane juice were selected: the amount of phosphoric acid added, the pre-ash pH value, and the neutralization pH value [7]. Inspect the indicators, each group of experiments were performed in parallel three times, and the average value was taken to reduce the error. A 3-factor 3-level experimental design scheme is used, and the orthogonal design experiment uses L9 (33), as shown in Table 2.

| Level | A amount of phosphoric acid added (ul/L) | B pre-ash pH value | C and PH value |
|-------|------------------------------------------|--------------------|----------------|
| 1     | 160                                      | 6.2                | 7.0            |
| 2     | 240                                      | 6.4                | 7.2            |
| 3     | 400                                      | 6.6                | 7.4            |

3. Test results and analysis

3.1. Single factor test results and analysis

| PAM (ppm) | Clarify the PH value | Turbidity/N TU | Refractive hammer/°Bx | Color value/IU |
|-----------|----------------------|----------------|------------------------|---------------|
| 2.0       | 7.3                  | 33             | 15.7                   | 1638          |
| 2.8       | 7.6                  | 32             | 16.0                   | 1494          |
| 3.0       | 7.6                  | 29             | 14.7                   | 1396          |
| 3.2       | 7.5                  | 30             | 15.5                   | 1527          |
| 4.0       | 7.6                  | 31             | 15.6                   | 1553          |

It can be seen from Table 3 that when the amount of PAM added is less than or higher than 3.0ppm, the overall effect of the clear juice is not good. When the amount of PAM added is 3.0ppm, the quality of the clear juice is better. The color value of the clear juice is 1396IU, the pH of the clear juice is 7.6, and the turbidity, 29NTU, refractive hammer of 14.7°Bx, close to the index of sulphurous acid clear juice. The clarification effect is better when the amount of PAM is 3.0ppm.
Table 4. Physicochemical indexes of clear juice with CTS

| CTS (ppm) | Clarify the PH value | Turbidity /NTU | Refractive hammer /° Bx | Color value/IU |
|-----------|---------------------|----------------|-------------------------|---------------|
| 2.0       | 7.7                 | 40             | 17.6                    | 1785          |
| 2.8       | 7.9                 | 40             | 17.4                    | 1781          |
| 3.0       | 7.8                 | 39             | 17.0                    | 1780          |
| 3.2       | 7.8                 | 51             | 17.3                    | 2041          |
| 4.0       | 7.7                 | 47             | 17.1                    | 2033          |

It can be seen from Table 4 that the color value of the clear juice after adding CTS is higher than 1780IU, the turbidity is higher than 39NTU, and the hammer is higher than 17.0° Bx, indicating that its solid solution content is large, non-sugar organic matter and pigment molecules are not completely flocculated, and the clear juice The quality is difficult to meet the sugar production requirements.

Table 5. Physical and chemical indexes of clear juice added with diatomaceous earth

| Diatomite (ppm) | Clarify the PH value | Turbidity /NTU | Refractive hammer /° Bx | Color value/IU |
|-----------------|---------------------|----------------|-------------------------|---------------|
| 2.0             | 7.0                 | 7.0            | 12.0                    | 1371          |
| 2.8             | 7.2                 | 7.2            | 11.6                    | 1296          |
| 3.0             | 7.2                 | 7.2            | 11.3                    | 1234          |
| 3.2             | 7.3                 | 7.3            | 11.8                    | 1387          |
| 4.0             | 7.7                 | 7.7            | 12.3                    | 1361          |

It can be seen from Table 5 that the color value of the clear juice after adding diatomaceous earth is about 1300IU, which is very close to the color value of the clear juice of the sugar factory sulfite process, and the turbidity is about 35NTU, which is slightly lower than that of the clear juice of the sugar plant sulfite process. However, the refractive index is about 12.0° Bx, which is much lower than the sulphurous acid method in the sugar factory. It shows that diatomaceous earth absorbs impurities while taking away larger sugars. Use alone will affect the sugar extraction rate.

The comprehensive analysis of the above three groups of single factor tests shows that PAM, CTS, and diatomaceous earth have their own advantages and disadvantages as sugar clarification aids, and they are not suitable to be used alone. These three materials are compounded and used as a composite flocculant to effectively reduce PAM The use of flocculant can give full play to the synergistic clarification effect of the flocculant. In the single-factor experiment, when the addition amount of PAM, CTS, and diatomaceous earth were all 3.0ppm, the clear juice color value was the lowest. Based on the synergistic effect of the compound clarifier, the median value of each flocculant addition was designed as 1.5ppm in the response surface test.
### 3.2. Response surface optimization test results and analysis

**Table 6. Response surface optimization test results**

| Number | Add amount (ppm) | Physical and chemical indicators of clear juice |
|--------|------------------|-----------------------------------------------|
|        | PAM  | CTS | Diatomite | After clarification PH value | Turbidity NTU | Refractive hammer (20°C)°Bx | Color value IU |
| 1      | 0    | 0   | 1.5       | 7.3                          | 36           | 16.1                        | 1630          |
| 2      | 3.0  | 0   | 1.5       | 7.5                          | 38           | 15.3                        | 1620          |
| 3      | 0    | 3.0 | 1.5       | 7.1                          | 33           | 15.7                        | 1610          |
| 4      | 3.0  | 3.0 | 1.5       | 7.3                          | 27           | 16.8                        | 1350          |
| 5      | 0    | 1.5 | 0         | 7.5                          | 26           | 15.3                        | 1153          |
| 6      | 3.0  | 1.5 | 0         | 7.6                          | 38           | 16.7                        | 1620          |
| 7      | 0    | 1.5 | 3.0       | 7.4                          | 45           | 16.5                        | 1534          |
| 8      | 3.0  | 1.5 | 3.0       | 7.5                          | 38           | 16.3                        | 1150          |
| 9      | 1.5  | 0   | 0         | 7.1                          | 39           | 15.7                        | 1689          |
| 10     | 1.5  | 3.0 | 0         | 7.6                          | 27           | 16.6                        | 780           |
| 11     | 1.5  | 0   | 3.0       | 7.5                          | 39           | 15.7                        | 1136          |
| 12     | 1.5  | 3.0 | 3.0       | 7.2                          | 36           | 16.8                        | 765           |
| 13     | 1.5  | 1.5 | 1.5       | 7.5                          | 26           | 16.3                        | 737           |
| 14     | 1.5  | 1.5 | 1.5       | 7.3                          | 22           | 14.7                        | 687           |
| 15     | 1.5  | 1.5 | 1.5       | 7.7                          | 28           | 16.5                        | 920           |
| 16     | 1.5  | 1.5 | 1.5       | 7.8                          | 37           | 17.3                        | 671           |
| 17     | 1.5  | 1.5 | 1.5       | 7.6                          | 51           | 16.8                        | 611           |

**Table 7. Response surface analysis of variance**

| Source of Variance | Sum of square | Degree of freedom | Mean square | F value | P value | Significance |
|--------------------|---------------|-------------------|-------------|---------|---------|--------------|
| Model              | 2.382E+006    | 9                 | 2.647E+005  | 8.33    | 0.0053  | **           |
| A                  | 4371.12       | 1                 | 4371.12     | 0.14    | 0.7216  |              |
| B                  | 3.081E+005    | 1                 | 3.081E+005  | 9.70    | 0.0170  | *            |
| C                  | 53956.12      | 1                 | 53956.12    | 1.70    | 0.2336  |              |
| AB                 | 15625.00      | 1                 | 15625.00    | 0.49    | 0.5057  |              |
| AC                 | 1.811E+005    | 1                 | 1.811E+005  | 5.70    | 0.0483  | *            |
| BC                 | 72361.00      | 1                 | 72361.00    | 2.28    | 0.1749  |              |
| A^2                | 1.271E+006    | 1                 | 1.271E+006  | 40.04   | 0.0004  | **           |
| B^2                | 3.249E+005    | 1                 | 3.249E+005  | 10.23   | 0.0151  | *            |
| C^2                | 33746.21      | 1                 | 33746.21    | 1.06    | 0.3369  |              |
| Residual           | 2.223E+005    | 7                 | 31753.22    |         |         |              |
| Error              | 1.668E+005    | 3                 | 55589.58    | 4.00    | 0.1067  |              |
| Pure error         | 55524.80      | 4                 | 13881.20    |         |         |              |
| Sum                | 2.604E+006    | 16                |             |         |         |              |

**Very significant difference (P < 0.01); *Significant difference (P< 0.05)**

It can be seen from Table 7 that the significance level of the model is P=0.0053, indicating that the model has reached the significance level. The lack of fit error value P=0.1067>0.05, indicating that the lack of fit is not significant, indicating that the regression equation fits well with the test and the model confidence is high.
Figure 1. The surface diagram of the interaction of various factors with the color value as the response value.

It can be seen from Figure 1 that there is a maximum stable point for the color value. The theoretically preferable material ratio of the composite flocculant predicted by the software Design-Expert 8.0.6 model is: PAM 1.67 ppm, CTS 1.91 ppm, and diatomaceous earth 2.08 ppm. The actual operation takes PAM 1.70 ppm, CTS 1.90 ppm, and diatomaceous earth 2.10 ppm as the material ratio of the composite flocculant. Under these conditions, three parallel verification tests are carried out. The average color value measured is 678 IU, which is 0.44 different from the predicted value of 681 IU, %, indicating that the selected model and factor level are highly reliable. The average color value of the final clear juice is 678 IU, the average PH value is 7.3, the average turbidity value is 34.5 NTU, and the average refractive hammer value is 16.2° Bx. Compared with the traditional sulfite method, the color value is reduced by about 50%, and it is turbid. The degree is reduced by about 10%, and the refractive hammer is equivalent to the PH value, indicating that the composite flocculant can absorb most of the suspended impurities and pigment molecules in the sedimentation of sugarcane juice, without causing excessive loss of sucrose, and is good for cleaning sugarcane juice.
3.3. Orthogonal test results and analysis

Table 8. Orthogonal test results and range analysis table

| Column | 1   | 2   | 3   | PH value of clear juice |
|--------|-----|-----|-----|-------------------------|
| Factor | A   | B   | C   |                          |
| 1      | 1   | 1   | 1   | 665                     |
| 2      | 1   | 2   | 2   | 970                     |
| 3      | 1   | 3   | 3   | 1849                    |
| 4      | 2   | 1   | 2   | 629                     |
| 5      | 2   | 2   | 3   | 1226                    |
| 6      | 2   | 3   | 1   | 611                     |
| 7      | 3   | 1   | 3   | 1242                    |
| 8      | 3   | 2   | 1   | 1096                    |
| 9      | 3   | 3   | 2   | 588                     |
| K₁     | 1161.333 | 845.333 | 790.667 |
| K₂     | 822.000    | 1097.333 | 729.000    |
| K₃     | 975.333    | 1016.000 | 1439.000    |
| R      | 339.333    | 252.000    | 710.000    |

It can be seen from Table 8 that the primary and secondary factors affecting the color value of the clear juice are C>A>B, and the optimal process level combination is A1B2C3. According to the optimal level, select the addition amount of phosphoric acid to be 160ul/L, the pre-ash pH value of 6.4, and the neutralization pH value of 7.4 to do three parallel tests and take the average value. The obtained clear juice is clear and bright, and the color value is low. The result is 612IU. The turbidity is 31NTU, the PH value is 7.3, and the hammerness is 16.4°Bx. Compared with the existing sulfite process clear juice, the color value and turbidity are greatly reduced, and the quality of the clear juice is improved. After adding the composite flocculant, the color value and turbidity of the clear juice are lower, and the solid solution content is within the normal range, indicating that the composite flocculant has a better clarification effect.

4. Conclusion
The cane juice cleaning process uses composite flocculants PAM 1.70 ppm, CTS 1.90 ppm, and diatomaceous earth 2.10 ppm to have a better clarification effect. Using less than the amount of PAM in the traditional process can also obtain good quality clear juice. The best process for using compound flocculants to clean cane juice is 160ul/L of phosphoric acid, pre-ash PH value of 6.4, and neutralizing PH value of 7.4. The resulting clear juice is clear and bright, with a color value of 612IU lower than that of traditional flocculants about 600IU and turbidity 31NTU is about 10NTU lower than traditional flocculants. The use of the composite flocculant combined with the cleaning process can improve the quality of the clear juice, and has good promotion and application value.

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