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Experimental Study on Liquid-solid Separation Process of Oxytetracycline Residue

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ABSTRACT
Traditional Chinese medicine (TCM) is an important part of traditional medicine in China. Ecological utilization of TCM residue is a hot research topic at present. The application of antibiotic residue is a difficult point in the study of resource utilization of drug residue in China. In this paper, four batches of harmless treated oxytetraamycin residue were taken as the research object. Filtration rate, turbidity of filtrate and moisture content of filter cake were taken as the assessment indexes, and the type of filter medium and filter pressure were taken as the investigation factors. DPS software was used to process the data, so as to obtain the optimal separation conditions.

Keywords: *Oxytetracycline residue, filtration rate, filtrate turbidity, filter cake moisture content, liquid-solid separation*

1. INTRODUCTION

Traditional Chinese medicine (TCM) is an important part of Chinese traditional medicine. With the progress of modern industrial science and technology, the industrialization and scale of traditional Chinese medicine production has been deepening, and the traditional Chinese medicine industry has been developing rapidly. At the same time, the waste of medicinal residue produced in the production and processing of Chinese herbal medicine is increasing day by day. Chinese medicinal residue is generally wet material, and it is easy to corrupt if it is not disposed for a long time. It has a strange odor and causes great pollution to the surrounding environment. A large number of studies have found that the general drug residue contains cellulose, protein, sugar, calcium, magnesium, iron, phosphorus and other substances, which have high usable value. Therefore, the ecological utilization of traditional Chinese medicine residue is a hot research topic at present.

The application of antibiotic residue is a difficult point in the study of resource utilization of drug residue in China.In the 1990s, there were reports of antibiotic residues being used directly as animal feed.But since 2002, China has classified antibiotic residues as "three industrial wastes" and banned their use as animal feed.In 2008, China defined antibiotic residue as hazardous waste, which needs to be recycled and treated by special departments, which is costly and a waste of resources.At present, the innocuous treatment process of oxytetracycline residue was studied, and a new innocuous treatment technology of antibiotic residue was explored. After innocuous treatment, the residue becomes a liquid-solid mixture system with larger viscosity and finer particles. In order to realize the effective utilization of the residue, liquid-solid separation is needed.

In this paper, four batches of harmless treated oxytetraamycin residue were taken as the research object. Filtration rate, turbidity of filtrate and moisture content of filter cake were taken as the assessment indexes, and the type of filter medium and filter pressure were taken as the investigation factors. DPS software was used to process the data, so as to obtain the optimal separation conditions.

2. TEST SECTION

The devices and test instruments used in the test are shown in Table 1.

2.1. Test equipment and instruments

Table 1 Test apparatus and instruments

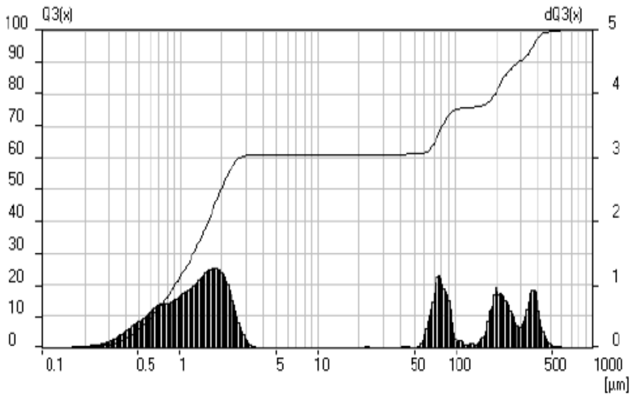
Device name	model	The manufacturer
Electronic balance	OHAUS ™	R.O.C Taiwan Co. Ltd
Filter separation unit	/	homemade
viscometer	NDJ-1	Shanghai Weighing Equipment Factory
Laser particle size meter	FrtischAnalysettle 22	German Frtisch Company
Digital display electric stirrer	JJ-1A	Jintan Hengfeng Instrument Manufacturing Co., Ltd
Super constant temperature tank	CH1015	Shanghai Sunny Hengping Scientific Instrument Co., Ltd
Electric heating constant temperature blast drying oven	DHG-9053A	Shanghai Yiheng Scientific Instrument Co., Ltd

2.2 Test materials

The test materials were provided by Lunan Pharmaceutical Factory. The initial temperature of the materials was 80℃, stored in a cool place and lowered to normal temperature, and then heated to 80℃ in a water bath before use.The basic physical property test results are shown in Table 2, and the particle size test results are shown in Figure 1.

Table 2 Basic property test of oxytetracycline residue

Material temperature /℃	Mass concentration of material	Viscosity（80℃）/cP	Density g/cm^3	PH
80	16.67%	800~1000	0.99	3



Mean	Values...						
D43 =	79.63 µm	D42 =	11.18 µm	D41 =	4.34 µm	D40 =	2.46 µm
D32 =	1.57 µm	D31 =	1.01 µm	D30 =	.77 µm		
D21 =	.65 µm	D20 =	.54 µm				
D10 =	.63 µm						

Fig. 1 Grain size distribution of oxytetracycline residue

2.3 Evaluation Indicators

There are three indicators to evaluate the effect of filtration and separation, including filtration rate, turbidity of filtrate and moisture content of filter cake.Where filtration rate refers to the time required to obtain the same filtrate volume;The turbidity of filtrate can be a qualitative characterization of the content of solid particles in the filtrate. The more the number of solid particles, the greater the turbidity of filtrate, and vice versa.The moisture content of filter cake refers to the percentage of water in the filter cake. The lower the moisture content of filter cake, the better the filtration effect, and the lower the drying load at the later stage.

3. TEST RESULTS AND DISCUSSION

3.1 Filter cake specific resistance and compressibility coefficient

The specific resistance and compressibility coefficient of filter cake are important indexes to evaluate the difficulty of material filtration and separation.

In order to further optimize the operating conditions of filtration and separation, the uniform design method was adopted, and U was selected$_6$*(6^4). The filter rate, filtrate turbidity and filter cake moisture content were taken as the assessment indexes, the filter medium model and filter pressure were taken as the investigation factors, and DPS software was selected as the data processing method. The original test data obtained were shown in Table 3. After preliminary processing of the test data in Table 3, the obtained results are shown in Table 4. It can be seen from the table that the average filtration rate and the moisture content of filter cake fluctuate little with the change of test conditions, while the turbidity of filtrate fluctuates greatly with the change of test conditions.

Table 3 Uniform design test data recording table

No.	Model of filter medium	Material quality /g	Material temperature /℃	filter area /cm^2	Filtration pressure /MPa	filter time	Mass of filter cake /g	Thickness of filter cake /mm	Humidity rate of filter cake	Volume of filtrate /mL	ilter cake per unit volume of material cm^3/mL
1	750AB8453	247.7	80	106	0.2	3’18”	238.7	10	63.28%	104	0.381
2	750AB8453	234.8	80	106	0.16	2’40”	113.9	10	65.68%	108	0.385
3	750AB9963	259.6	80	106	0.24	4’19”	125.8	10	63.38%	80	0.451
4	750AB9963	230.9	80	106	0.14	3’10”	120.1	10	63.75%	230.9	0.408
5	750AB9754	252.2	80	106	0.20	4’12”	106.6	10	63.78%	118	0.459
6	750AB9754	263.9	80	106	0.10	4’	106.3	10	62.35%	125	0.420

Table 4 Uniform design test results

Test no.	Factors affecting the		Assessment indicators		
	filter medium	Pressure/MPa	Average filtration rate /mL/s/cm^2/Y1	The filtrate turbidity Y2	The moisture content of filter cake is Y3
1	750AB8453	0.16	0.00476	17.3	63.28%
2	750AB8453	0.24	0.00590	39.0	65.68%
3	750AB9963	0.14	0.00398	19.5	63.38%
4	750AB9963	0.20	0.00497	28.1	63.75%
5	750AB9754	0.10	0.00419	9.9	63.78%
6	750AB9754	0.18	0.00552	12.8	62.35%

The experimental results were treated by quadratic polynomial stepwise regression method. Due to the existence of multiple assessment indexes in the test, the method of membership degree is adopted to process the data. The membership degree of filtration rate, turbidity of filtrate and moisture content of filter cake is calculated. The calculation formula is as follows:

$$Y_{1,\ i} = \frac{Y_i - Y_{min}}{Y_{max} - Y_{min}} = \frac{Y_i - 3.98}{5.90 - 3.98} = \frac{Y_i - 3.98}{1.92}$$

$$Y_{2,\ i} = \frac{Y_i - Y_{min}}{Y_{max} - Y_{min}} = \frac{Y_i - 0.0256}{0.1007 - 0.0256} = \frac{Y_i - 0.0256}{0.0751}$$

$$Y_{3,\ i} = \frac{Y_i - Y_{min}}{Y_{max} - Y_{min}} = \frac{Y_i - 1.522}{1.6038 - 1.5225} = \frac{Y_i - 1.5225}{0.0813}$$

According to the actual situation, the weights of filtration rate, filtrate turbidity and filter cake moisture content are 0.33, 0.33 and 0.33 respectively, so the comprehensive points are calculated as follows:

$$Y_i = 0.33Y_{1,\ i} + 0.33Y_{2,\ j} + 0.33Y_{3,\ j}$$

According to the above data processing method, the obtained results are shown in Table 5.Logical changes are made to the comprehensive score Y, namely$Y' = \ln(\frac{Y}{1-Y})$, and then carry out quadratic polynomial stepwise regression, and the fitting equation is as follows:

Y = 6.64895610-6.351557317 * $X_1$22.380243902 * X_2+ 1.2092 X X_1*X_1+ 13.185 X X_1*X_2

The significance analysis results of the equation are shown in Table 6. As can be seen from the table, the P value of the equation is 0.023418<0.05, and the P values of various regression coefficients are all less than 0.05. Therefore, the fitting equation has significance.

Table 5 Calculation results of membership degree of uniform design test

No.	factors		The evaluation index			membership			Comprehensive points Y
	X_1	X_2	Y1 filter rate	1/Y2 filtrate turbidity	1/Y3 Filter cake moisture	Y1 membership	1/Y2 membership	1/Y3 membership	
1	1	0.16	4.76	0.0579	1.5803	0.4063	0.4303	0.7107	0.5106
2	1	0.24	5.9	0.0256	1.5225	1.0000	0.0003	0.0004	0.3302
3	2	0.14	3.98	0.0512	1.5778	0.0000	0.3408	0.6800	0.3369
4	2	0.2	4.97	0.0355	1.5686	0.5156	0.1324	0.5674	0.4011
5	3	0.1	4.19	0.1007	1.5679	0.1094	0.9996	0.5583	0.5502
6	3	0.18	5.52	0.0783	1.6038	0.8021	0.7021	1.0006	0.8266

Table 6 Significance analysis table of fitting equation

			Analysis of variance table		
Sources of	Sum of squares	Degrees of	The mean	The F value	P values
Return to the	3.5996297	4	0.8999074	1025.2907	0.023418
residual	0.0008777	1	0.0008777		
The total	3.6005074	5			
	Regression	Standard	Partial	T value	P values
X1	6.351557	6.694658	0.999723	42.455258	0.0149923
X2	22.38024	1.281221	0.999346	27.640334	0.0230222
X1*X1	1.2092	5.1509068	0.9997328	43.248923	0.0147173
X1*X2	13.185	2.0706408	0.9996058	35.603666	0.017876
The complex correlation coefficient			The coefficient of determination R^2=0.999756		
Residual standard deviation					
Adjust correlation coefficient Ra=0.999390			Adjust the determination coefficient Ra^2=0.998781		

The best point predicted by the fitting equation is: Y '=2.599, X1=3, X2=0.24. The inverse logical transformation of Y' is carried out, and the Y value is obtained as 0.93. The verification test was carried out under this experimental condition, and the test results are shown in Table 7, from which we can see.

Table 7 confirmatory experimental results

	X_1	X_2	Y_1	Y_2	Y_3	Y	error
Forecast test	3	0.24				0.93	
Validation test	3	0.24					

3.2 Result discussion

We designed the method of uniform experiment, obtained the original experimental data, and used DPS software to process the data, and obtained the fitting equation with good significance. The optimal point of filtering oxytetracycline residue was determined as Y '=2.599, X1=3, X2=0.24. The verification test was carried out under this experimental condition, and the difference between it and the predicted value was small, indicating that the prediction effect of the fitting equation was good. This method is reliable and effective, and has certain application and popularization value.

4. CONCLUSION

In this paper, four batches of harmless treated oxytetraamycin residue were taken as the research object. Filtration rate, turbidity of filtrate and moisture content of filter cake were taken as the assessment indexes, and the type of filter medium and filter pressure were taken as the investigation factors. DPS software was used to process the data, so as to obtain the optimal separation conditions.

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