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Diatomaceous silica filter aid filtration for the effective separation of colloidal $Cr(OH)_3$ precipitate from tanning wastewater

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Abstract — Diatomaceous silica filter aid filtration was used in this study for the separation of colloidal Cr $(OH)_3$ precipitate from tanning wastewater. It was found that the addition of diatomaceous silica to tanning wastewater with pH>8. 2 can increase considerably the filtration rate of $Cr(OH)_3$ precipitate. The experimental results show that the average filtration rate(r, ml/s) is a function of diatomaceous silica content(d, g/100ml) in the wastewater, filtration temperature and pressure drop as well as area used. The relationship of r and d was found to be $r=0.06d+0.045$($d<16$g/100ml), over the range of filtration time of 0-270s used at 14℃, and filtration area of 0.0062m².

Keywords: diatomaceous silica; filter acid filtration; tanning wastewater; colloidal $Cr(OH)_3$ precipitate.

1 Introduction

Over the long run, every segment of an operation must prove itself economically to survive. The use of filter aids arose out of the need to improve the economics of the filtration unit operation. One of the first industries to feel the economic pinch caused by short filtration cycles was sugar refining, where the waxes from the sugar cane stalk plugged filters in short order. After considerable experimentation, it was found that crude diatomaceous silica, when mixed with raw sugar liquor, lengthened filtration cycles several fold while improving the quality of the filtered liquor.

The addition of filter aid to liquor can give porosity to the filter cake and provide a great number of microscopic holes for the liquid to flow through so that the filtration operation can be improved remarkably. While many different materials have been used or proposed as filter aids, only three have reached commercial importance. Of these, diatomaceous silica and perlite are of major importance while ground wood pulp is used in a number of specialty application where siliceous materials can not be used.

Diatomaceous silica is founds as sedimentary deposits and a unique natural material consisting of the skeletal remains of tiny organisms(diatoms). When added in proper amount to a liquid to be filtered, the resultant filter cake assumes the basic structure of the diatomaceous silica with ad-

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equate flow channels to keep the cycle going. The filter aids filtration has been used widely in industry and proved to be effective and economical.

Alkaline precipitation is unique approach for the removal of Cr(III) from tanning wastewater containing Cr(III). However, the resultant $Cr(OH)_3$ precipitate is colloidal, and very difficult to be filtered. The usual method for the separation of precipitate from wastewater is by plate-frame filtration process, which has many problems such as low filtration efficiency and difficulty to wash off large amount of organic substances from the precipitate.

In this paper, diatomaceous silica was used as filter aid for the filtration improvement in the separation process of $Cr(OH)_3$ precipitate from tanning wastewater. The filtration equation was proposed.

2 Experimental

Commercially available crude diatomaceous silica (Zhejiang, China) was used as filter aid. The tanning wastewater with a concentration of 1738 mg Cr (III)/L and pH 3.7 was kindly given by a tanning factory in Shanghai. It was found that it was more effective for the filtration improvement when diatomaceous silica was added to wastewater before the precipitation of Cr (III) by alkaline solution was conducted. The filtration experiments were carried out by adding a proper amount of diatomaceous silica (0-40g) to 100-200 ml wastewater, adjusting pH to about 9 while stirring, filtering while reading the total liquid volume (V, ml) for every 15 seconds under a constant pressure drop. Washing of the precipitate should be conducted before it was split. For filtration temperature experiment, the liquid with diatomaceous silica was heated in water bath, then added NaOH solution to pH 9, filtered immediately.

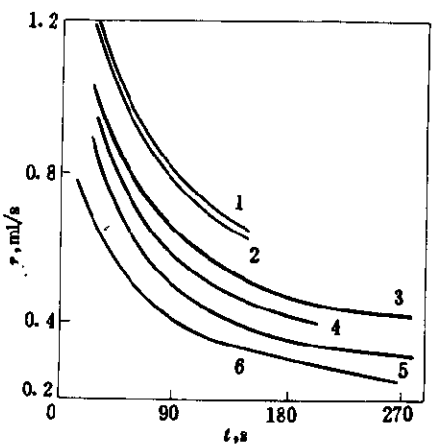

Fig. 1 The relationship between filtration rate and time under the condition of diatomaceous silica in liquid
Content of diatomaceous silica (g/100ml): 1—22; 2—16; 3—11; 4—8; 5—4; 6—0;
Pressure drop: 2.69 kPa; filtration area: 0.0062m²; 14℃;
r: filtration rate, ml/s; t: time, s

3 Results and discussion

3.1 Effect of diatomaceous silica content in liquid

The colloidal $Cr(OH)_3$ precipitate is very difficult to be filtered. However, the filtration operation can be improved considerably in terms of filtration rate (r, ml/s) when diatomaceous sili-

ca was added to the wastewater(Fig. 1). Morcover, the filtration rate was found to be linear to the content of diatomaceous silica in the liquid over the range of 0-16 g/100ml;

$$r = 0.06d + 0.045 (d < 16 g/100ml),$$

at a given time in the period of 0-150 seconds used (Fig. 2). The filtration rate increases slightly when the content of diatomaceous silica was higher than 16g/100ml.

3.2 Effect of filtration area

The increase of filtration area is always favorable to the filtration improvement in the process of solid - liquid separation. In the case of diatomaceous silica filter aid filtration of colloidal Cr $(OH)_3$ precipitate, the filtration rate was found to increase linearly with the increase of filtration area at given time(Fig. 3)

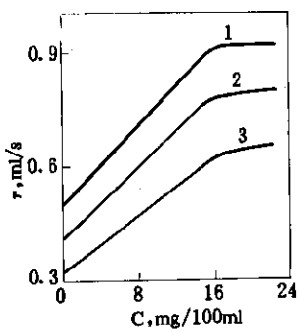

Fig. 2 The effect of content of diatomaceous silica in liquid on the filtration of $Cr(OH)_3$ precipitate at given time
Content of diatomaceous silica used(C):
0—22g/100ml in liquid; filtration time(s):1—60;
2—90; 3—150; filtration area: 0.0062m^2;
pressure drop:2.69kPa; 14℃;
r:filtration rate, ml/s

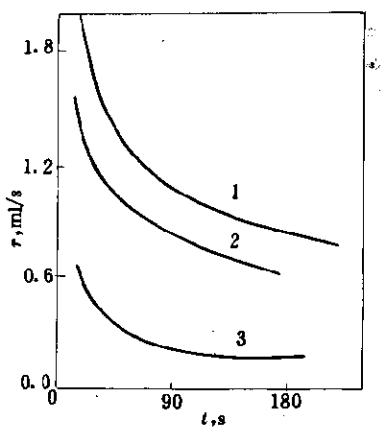

Fig. 3 The effect of filtration area on the diatomaceous silica filter aid filtration of $Cr(OH)_3$ precipitate
Filtration area (m^2): 1 — 0.01; 2 — 0.0062; 3 — 0.0015; pressure drop:2.69kPa; content of diatomaceous silica:15g/100ml, 14℃. Note: there exists a linear relationship between filtration rate(r,ml/s) and area (m^3) at a given time over the range of 0-180 s used(not shown in Fig. 3)

3.3 Relationship between filtration and pressure drop

The filtration rate increases as the increase of filtration pressure drop over the range of 0.9 — 2.69 kPa used. Moreover, the filtration rate with the presence of diatomaceous silica is always higher than that the absence of diatomaceous silica in the liquid even though the filtration pressure drop of the former is lower than that of the latter(Fig. 4).

3.4 Effect of liquid temperature

Diatomaceous silica was added to wastewater, adjusted pH to about 9, heated to the temper-

ature required and kept this temperature for 60 minutes, then filtered immediately. It was found that the higher the temperature was, the more effective the filtration can be obtained as shown in Table 1.

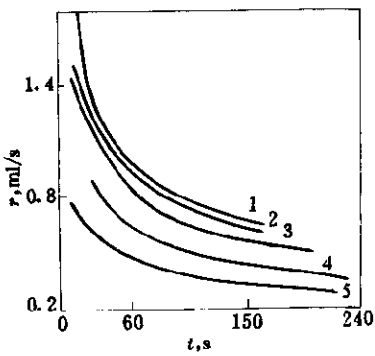

Fig. 4 The effect of pressure drop on the filtration
Pressure drop(kPa): 1—2.69; 2—2.32; 3—1.73; 4—0.90; 5—2.69; content of diatomaceous silica: 1-4: 15g/100ml; 5—0; filtration area: 0.0062m², 15℃; r: filtration rate, ml/s; t: time, s

Table 1 The relationship between the filtration and the initial liquid temperature

Filtration rate, *r*, ml/s	Temperature, ℃				
	80	75	65	50	14
15	4.0	2.8	1.9	1.7	1.5
30	3.0	2.4	1.7	1.6	1.2
Time, 45		2.1	1.6	1.4	1.0
s 60		1.7	1.4	1.3	0.9
75			1.3	1.2	0.8

Note: Filtration area: 0.0062m²; pressure drop: 2.69kPa; content of diatomaceous silica: 20g/100ml

3.5 Washing of the cake

It is impossible to wash off the soluble organic substance from the colloidal $Cr(OH)_3$ precipitate cake alone. However, the washing of cake was much more easily when the cake contained diatomaceous silica as shown in Table 2. If the liquid was heated, the washing rate of resultant cake increases considerably (Table 3). It may be considered that the addition of diatomaceous silica to liquid makes the washing of cake possible and effective.

Table 2 The influence of diatomaceous silica content on the washing of cake

Content of diatomaceous silica, g/100ml	0	4	8	12	16	22
Washing rate, ml/s	0.10	0.15	0.20	0.25	0.34	0.25

Note: Temperature: 14℃; filtration area: 0.0062m²; pressure drop: 2.69kPa

Table 3 Effect of initial liquid temperature on the washing of resultant cake

Washing rate, ml/s		Initial liquid temperature, ℃				
		14	50	65	90	70
Content of diatomaceous silica, g/100ml	0	0.10				0.20
	20	0.26	0.50	0.76	0.90	0.80

Note: The temperature of both the cake and the water for washing was 14℃; washing area: 0.0062m²; pressure drop: 2.69kPa

3.6 Leaching of $Cr(OH)_3$ from the cake

The experimental results show that the $Cr(OH)_3$ in cake can be leached quantitatively with H_2SO_4 solution. Filtered, and both the aqueous solution and diatomaceous silica can be reused in the next process.

3.7 Proposal of filtration equation

The filter aid filtration of diatomaceous silica is typically a cake filtration process. In this work, the filtration pressure drop was kept at 2.69kPa. It was found that a plot of the logarithm of total liquid volume flowed through the cake, v(ml), vs. the logarithm of time, t (s), will result in a straight line. The slope of the lines is dependent of the content of diatomaceous silica in the wastewater over the range of filtration time studied(Table 4).

Table 4 Filtration equation of diatomaceous silica filter aid filtration for the solid-liquid separation of colloidal $Cr(OH)_3$ precipitate from tanning wastewater

No.	Content of DS, d. g/100ml	Straight line of $\lg v-\lg t$ $\lg v=k\lg t+1$		Expression of filtration equation, $0<t\leqslant150$
		K	I	
0	0	0.632	0.345	$V_0=2.21t^{0.632}$
1	4	0.537	0.600	$V_1=3.98t^{0.537}$
2	8	0.588	0.567	$V_2=3.69t^{0.588}$
3	11	0.476	0.762	$V_3=5.78t^{0.476}$
4	16	0.641	0.595	$V_4=3.93t^{0.64}$
5	20	0.583	0.668	$V_5=4.66t^{0.583}$

Note: The experimental conditions are the same as shown in Fig. 1. DS=diatomaceous silica

By simple calculation it can be found that at any filtration time the filtration rate of V_4 is always higher than that of the other:

$$V_1<V_2<V_3<V_4>V_5 \quad , \qquad 0<t<55$$

or:

$$\begin{cases} V_1<V_2>V_3<V_4>V_5 \\ V_4>V_2 \end{cases} \qquad 55<t<150$$

It may be considered from the experimental results that the optimum content of diatomaceous silica was about 16g/100ml.

Theoretically, a plot of the $\lg v-\lg t$ should result in a straight line having a slope of 0.5 for a constant pressure filtration(Philip, 1988). In the case of diatomaceous silica filter aid filtration, the plot of $\lg v-\lg t$ is indeed a straight line. However, the slope of lines is not 0.5 but over the range of 0.47-0.65(Table 4). Obviously, the cake of $Cr(OH)_3$ and diatomaceous silica is not absolutely hard and incompressible. Moreover, the medium resistance is appreciable and can not be negligible. These factors will make the slope of $\lg v-\lg t$ deviate from the theoretical value. The plot of $\lg v-\lg t$ for $Cr(OH)_3$ precipitate alone has also a straight line with a slope of 0.632, which is very similar to that for diatomaceous silica filter aid filtration behavior. It may be pro-

posed that once the filtration operation starts, the fine solid particles of colloidal $Cr(OH)_3$ precipitate deposit on the filter cake, they remain in place without migration. So the filtration of Cr $(OH)_3$ precipitate is similar to that of hard and incompressible solid precipitate. However, the particles of $Cr(OH)_3$ precipitate is so fine that they plug filters in short order and give smaller and smaller porosity to the cake, resulting in difficult filtration process.

Diatomaceous silica filter aid filtration is an unique approach for the separation of colloidal precipitate because of its commercial availability and low price. In this work, only the laboratory scale test was conducted. From the experimental results it can be considered that the diatomaceous silica filter aid filtration is favorable to the separation of colloidal $Cr(OH)_3$ precipitate and has potential significant application in industry.

It may be concluded that the addition of diatomaceous silica filter aid can improve considerably the filtration of $Cr(OH)_3$ precipitate from the mother liquor. The filtration equation with a straight line of $\lg v - \lg t$ plot supposed that the filter aid filtration can be approximately by constant pressure filtration process of hard and incompressible cake.

References

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