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Research Article

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Studies on purification of platycodins by AB-8 macroreticular resin

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ABSTRACT

To purify the platycodins by AB-8 macroporous resin, the adsorption isotherms and adsorption kinetics were studied. The effect of loading sample process on breakthrough curves of platycodins in AB-8 resin fixed-bed was also investigated. The result show that the concentration of loading sample was 2.0 mg/mL, the elution velocity of flow was 2.0 mL/min and the concentration of eluant was 80% ethanol. The product purity was 92.13% with a recovery rate of 78.41% on this condition. The research results provided a suitable method for puritying platycodins.

Key words: AB-8 macroreticular resin, Purification, Platycodins

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INTRODUCTION

The root of *Platycodon grandiflorum* (Jacq.) A. DC.(Campanulaceae) has many function , such as anti-ulcer, antitussive and hypolipidemic [1]. Triterpenoid saponins are the main active constituents of it[2–6]. Currently, many scholars have used macroporous resins to separate and purify platycodins. Xianwen Yang [7-8] prepared platycodins by by means of ZTC-1 macroporous adsorption resin. Mingxia Liand prepared platycodins by LSA230 macroporous adsorption resin. AB-8 macroreticular resin was also a better macroporous adsorption resin for preparation platycodins. So, In this paper, AB-8 macroreticular resin was used to separate and purify platycodins from *Platycodon grandiflorum* (Jacq.) A. DC root.

EXPERIMENTAL SECTION

1.Materials and reagents

Platycodon grandiflorum root was purchased from Huainan Dayaofang Co., Ltd. (China). AB-8 macroreticular resin was obtained from Cangzhou Baoen adsorbing material United Win Technology Limited (China). Others reagent (chemical grade) were obtained from Huainan Dayaofang Co., Ltd.

2.Instruments

Ultraviolet and visible spectrophotometer , Beijing Pgeneral Co., Ltd. China. Temperature oscillation incubator , aicang experimental facilities factory, China. Column cromatographic system , Shanghai Kanghua biochemistry apparatus factory.

3.Preparation of the extract of crude platycodins

Platycodon grandiflorum powder mixed with ethanol (70%, V/V) at ratio of solid and liquid 1:8 g/mL. Then were extracted using ultrasonic wave at 200W for 45mins by. Then the extracts was centrifuged , evaporated and dried.

4. Determination of platycodins

Platycodins was determined by vanillin-perchloric acid spectrophotometry accords Xu et al[9]methods.

5. Adsorption equilibrium and desorption experiments

Extracts solution with certain platycodins concentration mixed with resins, shaking at 25±1 ℃, 150 r/min for 24h to establish equilibrium. Then the amount of platycodins left in the solution was analyzed using spectrophotometer at 475 nm. The similar experiments were performed at various temperatures (20 ℃, 30 ℃ ,40 ℃). The adsorption capacity and adsorption ratio of the resins, Q_1(mg/g) and E_1(%), respectively, were calculated according to

$$Q_1 = \frac{C_0V_0 - C_1V_1}{W} \qquad E_1(\%) = \frac{C_0V_0 - C_1V_1}{C_0V_0} \times 100\%$$

where C_0 and C_1 were the initial and equilibrium liquid-phase concentrations (mg/mL), respectively, V_0 and V_1 were the volume of the initial and after adsorption solution (mL) , respectively, and W was the weight of wet resins (g).

After finishing adsorption equilibrium, the appropriate volume of 70% ethanol was added to the adsorbents, shaking the solution at 25±1 ℃ for 24 h, and measuring the desorption concentration in the solution. The desorption capacity and desorption ratio of the rein, Q_2 (mg/g) and E2(%), respectively, were calculated according to

$$Q_2 = \frac{C_2V_2}{W} \qquad E_2(\%) = \frac{C_2V_2}{C_0V_0 - C_1V_1} \times 100\%$$

where C_2 was the platycodin desorption solution concentration (mg/mL), and V_2 was the volume of desorption solution (mL).

6. Fixed-bed adsorption and desorption

Adsorption breakthrough curve was determined by fixed-bed experiments using a glass column packed with resin particles. The velocity of flow was regulated by a precision pump. The solution was introduced downward into the column. Desorption of platycodins from the sorbents was performed using ethanol with different concentrations. Samples were withdrawn from the effluent line and analyzed by the spectrophotometer at 475 nm.

RESULTS AND DISCUSSION

1. Static adsorption dynamics of platycodins in aqueous solution

Fig.1 displays the adsorption dynamic curve of platycodin adsorbed onto AB-8 macroreticular resin at different temperatures, and the required time from the beginning to the equilibrium is about 300 min. The Lagergren rate equation ln(qe-q)=lnqe-kt and Bangham rate equation q=kt/m are employed to fit the adsorption dynamic data. Where q is the adsorption capacity of platycodins at the contact time t (mg/g), k is adsorption rate constant [g/(mg. min)], and m is constant, respectively.

The fitted results (Tab.1) indicate that the adsorption obey the pseudo-first-order rate equation Lagergren. The pseudo-first-order rate equation means that the reaction rate is only in relation to the simple equation of the substantial concentration. In the adsorption process, the adsorption takes place in the solid/liquid surface. The liquid phase is the platycodins solution, and the solid phase is the AB-8 macroreticular resin. The fitted results imply that the adsorption has something to do with the liquid phase solution.

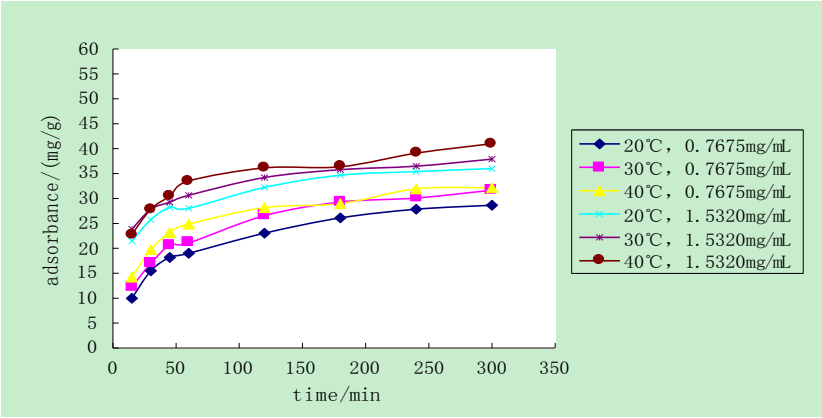

Fig.1 Static adsorption dynamic curve

Tab.1 The fitting results of kinetic equations

		Lagergren equation		Bangham equation	
T(K)	C(mg/mL)	k(min^{-1})	R^2	k(min^{-1})	R^2
293	0.7675	0.0137	0.9788	5.5134	0.9709
303	0.7675	0.0117	0.9884	6.69	0.9716
313	0.7675	0.0209	0.9731	9.194	0.9431
293	1.532	0.0139	0.9796	14.6342	0.9762
303	1.532	0.0145	0.9871	16.7725	0.9866
313	1.532	0.0197	0.9587	15.7057	0.9532

2. The adsorption isotherms of platycodins on AB-8 resin

Fig.2 shows that equilibrium adsorption capacity for platycodins increase by equilibrium concentration increasing. Based on isothermal adsorption models[10, 11] of Freundlich $\ln q_e=\ln K_f+1/n \ln C_e$ (n describes changing trend of isotherms, K_f reflectes adsorption quantity) and Langmuir $1/q_e=1/q_m+1/K_LC_e$ (q_m is single molecular layer saturated absorption, K_L is adsorption coefficient), the adsorption isotherms of platycodins are fitted.

From Tab.2, it can be perceived Freundlich model is more appropriate than Langmuir model.

Tab.2 The fitting parameters of isotherms equation

	Langmuir model			Freundlich model		
T(K)	q_m	K_L	R^2	K_f	1/n	R^2
293	34.83148	2.4539	0.9987	24.41727	0.34415	0.9607
303	34.07549	2.99262	0.9979	23.10461	0.40276	0.9722
313	32.2449	2.11646	0.9967	21.45446	0.442	0.9898

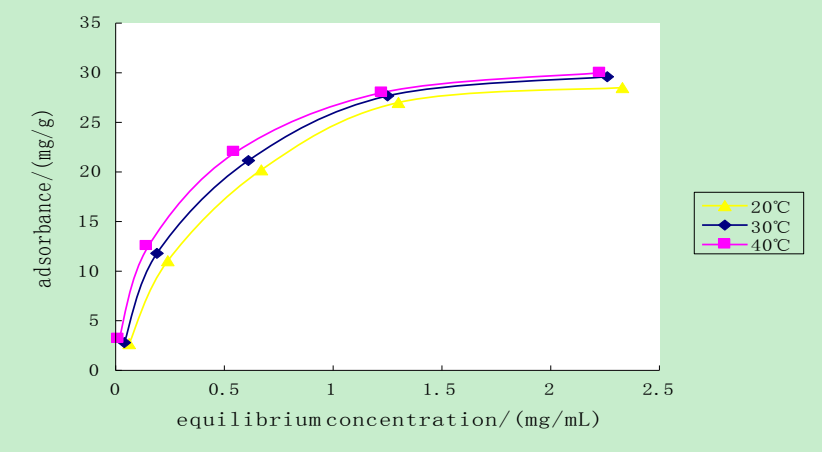

Fig.2 The adsorption isotherms

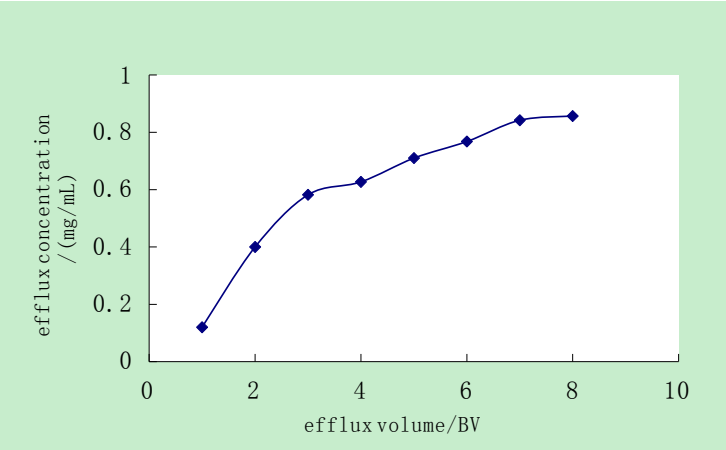

Fig.3 The adsorption breakthrough curve of AB-8 resin

3. The breakthrough curve of AB-8 resin

Fig.3 shows a typical concentration history of the effluent for a fixed-bed adsorption. The breakthrough volume for 0.12 mg/mL exit platycodins concentration is calculated to be 1BV, for 2.5 mL/min flow rate for an inlet platycodins

concentration of 2.5 mg/mL. The concentration of platycodins solution is stable with 7BV effluent volume. The saturated adsorption is obtained to be 213.75 mg, and the dynamic adsorption capacity is calculated to be 28.5 mg/mL.

4. Effect of loading concentration on adsorption results

Effect of loading concentration on adsorption rate was in fig.4. The results show original concentration of platycodins aqueous solution affects adsorption quantity. The adsorption quantity changes with original concentration. With the original concentration increasing, the adsorption rate of platycodins by macroreticular resin increases, when the concentration is up to 2.0 mg/mL, the adsorption rate is at its maximum. However, the adsorption rate is on a declining curve with a continuous augment of original concentration, Maybe there is a leak in the resin column leading to a fall in handling capacity of sample. So we take 2.0mg/mL as the optimum concentration.

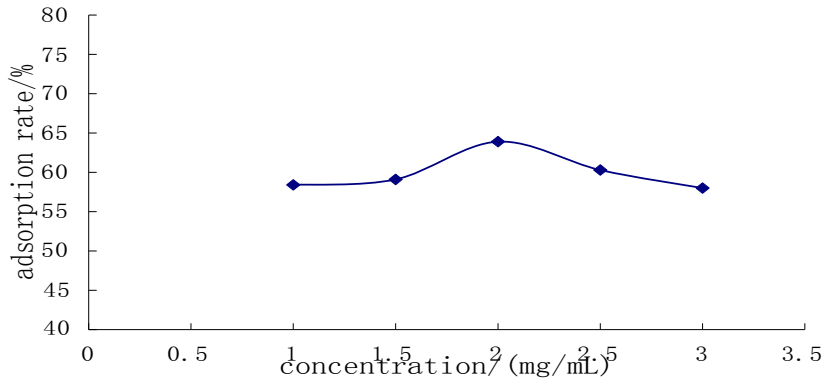

Fig.4 Effect of loading concentration on adsorption rate

5. The desorbent and design of the elution curve

In this study, 80% ethanol is used as a desorbent for platycodins. Fig.5 shows us flow rate on the desorption behavior. The elution peak is getting narrower and narrower with increasing elution rate, which implies the separating effect is obvious. Yet too quick elution rate results in waste of ethanol. So 2.0 mL/min flow rate is taken to carry the following experiments.

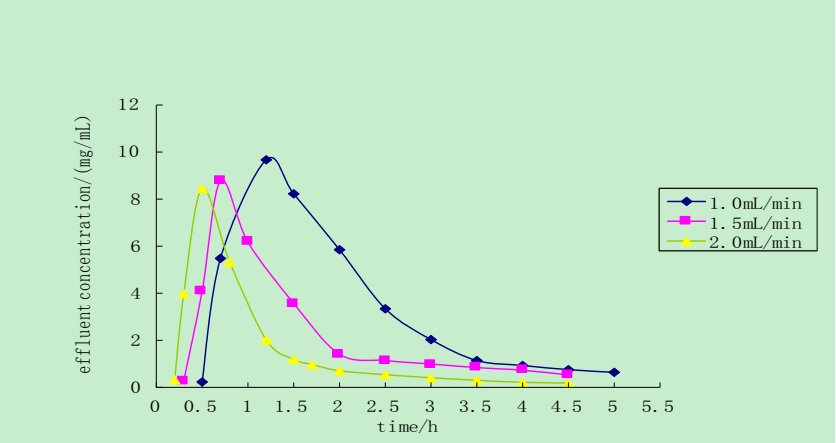

Fig.9 Effect of elution rate on the elution curve

6. Demonstration experiment

The resin column is loaded for 2.5 mL/min flow rate for an inlet platycodins concentration of 2.5mg/mL and is eluted by 8BV aqua destillata after equilibration to remove redundant platycodins and other hydrosoluble impurities, then the ethanol of 80% is used as eluent to elute the resin for 2.0 mL/min flow rate. The effluent is collected, evaporated, concentrated and dried to powder of platycodins. Finally, we obtain the platycodins product with the purity of 92.13% and the recovery of 78.41%, which shows it is feasible to use the purification technology.

CONCLUSION

The dynamic adsorption and desorption process were analysed and the results were as follow: the dynamic adsorption capacity was 28.5 mg/mL; the optimum loading concentration was 2.0 mg/mL. The ethanol of 80% was used as eluent to elute the resin for 2.0mL/min flow rate. In this condition, we obtained the platycodins product with the purity of 92.13% and the recovery rate of 78.41%, which showed it was feasible and had potential application value in industry.

Acknowledgements
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