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Enrichment and purification of gardenia yellow from *Gardenia jasminoides* var. *radicans Makino* by column chromatography technique

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ABSTRACT

In present study, the performance and separation characteristics of nine macroporous resins for the enrichment and purification of gardenia yellow from *Gardenia jasminoides* var. *radicans Makino* have been evaluated. The adsorption and desorption properties of crude gardenia yellow solution on macroporous resins including HPD722, HPD100, HPD100A, HPD400, HPD400A, D101, AB-8, XAD-16, and NKA-9 have been compared. Then, HPD722 was chosen to purify gardenia yellow because of its strong adsorption and desorption abilities as well as high selectivity. Column packed with HPD722 resin was used to perform dynamic adsorption and desorption tests to optimize the separation process of gardenia yellow. The optimal conditions were as follows: The crude gardenia yellow solution with concentration of 15 mg/mL was loaded in column packed with HPD722 resin at the flow rate of 1.0 mL/min, and the adsorbate-laden column was washed with 800 mL water, 600 mL 15% ethanol water solution respectively at the speed of 2.5 mL/min, then desorbed with 200 mL 80% ethanol water solution at the speed of 3.5 mL/min. The colority of the product obtained were up to 300. The method developed in this study provides a new approach for scale-up separation and purification of gardenia yellow from *G. jasminoides* var. *radicans Makino*.

1. Introduction

Gardenia fruit, the dried ripe fruit of *Gardenia jasminoides Ellis* (Rubiaceae), is a traditional Chinese medicine widely used orally as an antiphlogistic, choleretic, diuretic, hemostatic and anticancer agent, and externally as a resolutive in treatment of sprains and bruises [1]. In addition, gardenia yellow extracted from this fruit is widely used in food processing as a rare natural water-soluble yellow pigment. Because of its water solubility, it has been used as a natural food colourant for a long time in Japan, mainly in coloured juice, jelly, candy and noodles [2]. It also has advantages such as high stability, strong tinting strength, of nutrition and health-keeping value compared with many other natural food colorants. These properties, combined with the worldwide trend toward replacing synthetic colorants with natural pigments, have generated a growing demand for gardenia yellow in international markets, especially in Japan.

The studies of extraction and purification of gardenia yellow mostly focus on *G. jasminoides Ellis.* The gardenia yellow was usually extracted from *G. jasminoides Ellis* by methods such as water extraction [3], alcoholic extraction [4] and ultrasonic extraction [5]. Gardenia yellow is a mixture and its main components include crocin, crocetin, geniposide, genipin [6] in which crocin occur as the main pigment [7]. Gardenia yellow is easy to fade and turn green because of the coexistence of geniposide and chlorogenic acid in the crude gardenia yellow extraction. Thus, an efficient method to remove geniposide and chlorogenic acid from gardenia yellow is necessary. As so far, the methods for the purification of gardenia yellow from *G. jasminoides Ellis* include CO_2-SFE [8], gel chromatography [9], ultra filtration [10] and adsorption method [11,12], in which only adsorption method could really be scaled-up for industrial production. There has been a growing interest in employing macroporous resins to separate the bioactive compounds from crude extracts of herbal raw materials; this was due to the unique advantageous adsorption properties including ideal pore structure and various surface functional groups available on these resins, in addition to low operation costs, less solvent consumption and easy regeneration of these resins [13,14].

Gardenia yellow exists in different species of *Gardenia jasminoides* and its content varies. Gardenia genus fruit is a nature plant rich in crocin besides saffron. *Gardenia jasminoides* var. *radicans*

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Table 1
Physical properties of macroporous resins.

Name	Polarity	Particle diameter (mm)	Average pore diameter (Å)	Surface area (m^2/g)
D101	None-polar	0.25–0.85	90–100	500–550
HPD100	None-polar	0.3–1.25	85–90	650–700
HPD100A	None-polar	0.25–0.85	95–100	650–700
HPD722	Weak-polar	0.3–1.25	130–140	485–530
XAD-16	Weak-polar	0.7	150	800
AB-8	Weak-polar	0.3–1.25	130–140	480–520
HPD400	Moderate-polar	0.3–1.2	75–80	500–550
HPD400A	Moderate-polar	0.25–0.85	85–90	500–550
NKA-9	Polar	0.3–1.25	155–165	250–290

Makino, also named *Gardenia jasminoides* var. *grandiflora* is the dried fruit of *Gardenia jasminoides Ellis* var. *grandiflora*, which is a form of *G. jasminoides Ellis*. And it has been reported that content of gardenia yellow in *G. jasminoides* var. *radicans Makino* was much higher than that in *G. jasminoides Ellis* [15]. As the source of gardenia yellow, *G. jasminoides* var. *radicans Makino* has advantages including higher content, lower cost and higher yield compared with *G. jasminoides Ellis*. However, to our knowledge, few reports on the extraction and purification of gardenia yellow from *G. jasminoides* var. *radicans Makino* have been published to date.

This study aims to develop an efficient method for the enrichment and purification of gardenia yellow from *G. jasminoides* var. *radicans Makino*. The results in this study are significant in industrial production of gardenia yellow from *G. jasminoides* var. *radicans Makino*.

2. Materials and methods

2.1. Chemicals and reagents

Analytical-grade ethanol, hydrochloric acid, sodium hydroxide were purchased from Damao Chemical Reagent Factory (Tianjin, China). HPLC-grade methanol was obtained from YongDa Chemical Reagent Co., Ltd. (Tianjin, China). All aqueous solutions were prepared with distilled water. The standard sample of crocin-1 ($C_{44}H_{64}O_{24}$, ≥98% purity) was obtained from Sigma. *G. jasminoides* var. *radicans Makino* was purchased from Zhangshu city, Jiangxi province, China.

2.2. Adsorbents

Macroporous resins including HPD722, HPD100, HPD100A, HPD400 and HPD400A were purchased from Cang Zhou Bon Adsorber Technology Co., Ltd. (Hebei, China), AB-8 and NKA-9 from The Chemical Plant of Nankai University (Tianjin, China), D101 from Anhui Sanxing Resin Technology Co., Ltd. (Anhui, China) and XAD-16 from Rohm & Haas (American). Their physical properties are summarized in Table 1.

The resins were soaked in ethanol, shaken for 24 h before filtration. Then the resins were pre-treated with 5% HCl and 2% NaOH solutions in succession to remove the monomers and porogenic agents trapped inside the pores during the synthesis process, and then were subsequently washed to neutrality with distilled water before use.

2.3. Preparation of Gardenia jasminoides var. radicans Makino extracts

The powdered sample (90 g) of *G. jasminoides* var. *radicans Makino* was extracted by 720 mL ethanol–water (50:50, v/v) solution at 60 °C for 1 h. The residue was further extracted with the same method. The combined aqueous extracts were purified by membrane filtration and then concentrated to paste-like in a rotary evaporator under reduced pressure at 50 °C. Then the concentrated solution was stored in fridge at 4 °C.

2.4. Measurement of concentration of gardenia yellow

The concentration of gardenia yellow was measured by spectrophotometry. The calibration curve was plotted using the absorbance at 440 nm as the abscissa and the corresponding concentration of gardenia yellow in mg/mL as the ordinate. The linear regression equation was $C = 0.052A - 0.0009$ ($R^2 = 0.9993$), and its linear range was between 0.0072 mg/mL and 0.036 mg/mL.

2.5. Static adsorption and desorption tests

The static adsorption and desorption tests of gardenia yellow on macroporous resins were performed as follows: 1 g samples of hydrated test resins together with 50 mL aqueous solution of crude gardenia yellow (4.28 mg/mL) were added into a flask, then shaken (125 rpm) at 25 °C for 24 h. After adsorption equilibrium was reached, the resins were desorbed with 50 mL ethanol–water (80:20, v/v) solution, then shaken (125 rpm) at 25 °C for 20 h. The concentrations of gardenia yellow in the sample solution after adsorption of a certain time were monitored at different time intervals till equilibration to get adsorption kinetic curves.

The selectivity of resins was based on the capacities of adsorption, the ratios of adsorption and desorption and the OD value (A_{238}/A_{440}). Maximum absorption wavelengths of geniposide (main impurity of gardenia yellow) and crocin (main components of gardenia yellow) are 238 nm and 440 nm, respectively.

The capacity of adsorption, the ratios of adsorption and desorption as well as OD value were quantified as the following equations:

• Adsorption capacity:

$$Q_e = \frac{(C_0 - C_e)V_i}{m} \quad (1)$$

• Adsorption ratio:

$$E(\%) = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

where Q_e is the adsorption capacity, which represents the mass of the analyte adsorbed on 1 g wet resin at the adsorption equilibrium (mg/g); E is the adsorption ratio (%), which means percentage of total analyte being adsorbed at the adsorption equilibrium; C_0 and C_e are the initial and equilibrium concentration of gardenia yellow in the solutions (mg/mL), respectively; V_i is the volume of the initial sample solution added into the flask (mL); m is the weight of resin (g).

• Desorption ratio:

$$D(\%) = \frac{C_d V_d}{Q_e} \times 100 \quad (3)$$

•OD value:

$$\mathrm{OD} = \frac{A_{238}}{A_{440}} \quad (4)$$

where D is the desorption ratio (%); C_d is the concentration of the solutes in the desorption solutions (mg/mL); V_d is the volume of the desorption solution (mL); Q_e is the same as that defined above. OD means the OD value, and A_{238}, A_{440} are the absorbances at 238 nm, 440 nm (A_{238}, A_{440}) of the desorbed solution, respectively.

2.6. Dynamic adsorption and desorption

Dynamic adsorption and desorption experiments for gardenia yellow were carried out on glass columns (2.6 cm × 30 cm) wet-packed with 25 g (wet resin) of the selected hydrated resin. The bed volume (BV) of resin was 85 mL. After the sample loading (200 mL), the adsorbate-laden column was washed first by distilled water, and then eluted with ethanol–water (15:85, v/v) and ethanol–water (80:20, v/v), respectively. The conditions including concentration of the loading sample solution (5, 10, 15, 20 mg/mL), sample flow rate (0.5, 1.0, 1.5, 2.0 mL/min), desorption solvent (ethanol–water 30:70, 80:20, 90:10, v/v), desorption flow rate (1.5, 2.5, 3.5, 5.0 mL/min) and amounts of different eluents for the purification of gardenia yellow were optimized.

2.7. Measurement of the colority of gardenia yellow

The colority of gardenia yellow was measured following the Chinese national standard entitled GB7912-2010 [16]. The concrete steps were as follows: Firstly, 0.15 g powdered sample was dissolved in water. Then the solution was transferred to a 100 mL volumetric flask and brought to volume and shaken. Next, 10 mL sample solution was transferred to another 100 mL volumetric flask, brought to volume and shaken, too. Finally, the absorbance A of the diluted gardenia yellow solution was measured in a standard 1.0 cm pathlength cuvette by spectrophotometer at the maximum absorption wavelength 440 nm (absorbance should be controlled between 0.3 and 0.7). The colority of gardenia yellow can be calculated as the following equation:

$$E = \frac{A}{C} \times \frac{1}{100} \quad (5)$$

where E stands for the colority, A is the absorbance at 440 nm, and C is the concentration of the diluted gardenia yellow solution (g/mL).

2.8. UV–vis analysis of gardenia yellow before and after purification

Gardenia yellow solutions before and after purification were scanned between 200 and 700 nm using ultraviolet and visible spectrophotometry.

2.9. HPLC analysis of gardenia yellow [17]

Waters 2695-2996 liquid chromatographic system was used to determine the purity of gardenia yellow. Chromatographic separation was carried out by Kromasil C_{18} column (4.6 mm × 250 mm, 5 μm). The mobile phase was methanol–water (50:50, v/v); the flow rate was 1.0 mL/min; the column temperature was maintained at 25 °C; the separated gardenia yellow was detected at a wavelength of 440 nm and the injection volume was 20 μL.

3. Results and discussion

3.1. Adsorption capacity, ratios of adsorption and desorption as well as OD value

As shown in Table 2, all the resins in this study showed high adsorption and desorption capacity except NKA-9. In general, the adsorption ability of resin is correlated with its surface area and the chemical features of the adsorbed substance. Resins with weak-polarity showed stronger adsorption ability to weak-polar substances such as crocin and crocetin compared with non-polar, moderate-polar and polar resins. We have observed that the HPD722 and XAD16 have higher adsorption capacity for the gardenia yellow (192.42 mg/g for HPD722 resin; 191.20 mg/g for XAD16 resin, respectively). In addition, we noted that the non-polar resin HPD100 showed better adsorption capacity for gardenia yellow (198.39 mg/g) because HPD100 resin had a relative larger surface area. However, the desorption ratio of HPD100 resin was lower than those of HPD722 and XAD16 resins owing to its relative smaller average pore diameters (82.26% for HPD100 resin, 85.91% for HPD722 resin, 85.99% for XAD16 resin, respectively). OD value could reflect the selectivity of resin on gardenia yellow and geniposide in a degree. The lower the OD value is, the higher the selectivity of resin is for gardenia yellow and geniposide. A quick perusal of Table 2 indicated that the OD value of HPD722 resin was the lowest among HPD100, HPD722 and XAD16 resins. Considering the measured adsorption capacity, the ratios of adsorption and desorption, as well as the OD value, we selected the HPD722 resin to perform the further experiment.

Table 2
Adsorption capacity, adsorption and desorption ratio and adsorption selectivity of different resins on gardenia yellow.

Resin	Adsorption capacity (mg/g)	Adsorption ratio (%)	Desorption ratio (%)	OD value (A_{238}/A_{440})
HPD100	198.39	92.77	82.26	0.84
HPD722	192.42	89.98	85.91	0.78
XAD16	191.20	89.41	85.99	1.04
D101	190.34	89.00	79.37	0.79
HPD400	188.84	88.30	85.45	0.75
HPD100A	187.52	87.72	81.09	0.79
AB-8	184.73	86.38	78.75	0.77
HPD400A	184.41	86.23	82.28	0.83
NKA-9	94.89	44.37	72.06	0.85

3.2. The adsorption kinetics curve for gardenia yellow on HPD722 resin

Adsorption kinetics curve was obtained for gardenia yellow on HPD722 resin. As shown in Fig. 1, the adsorption ratio of the HPD722 resin increased rapidly in the first 2 h, then followed by a slow increase until it reached equilibrium after 6 h. The fast adsorption process of the HPD722 resin in the first 2 h was due to the high diffusivity of gardenia yellow into micropores of the resin, and the slow adsorption process of HPD722 resin 2 h later was due to the high intraparticle mass transfer resistance.

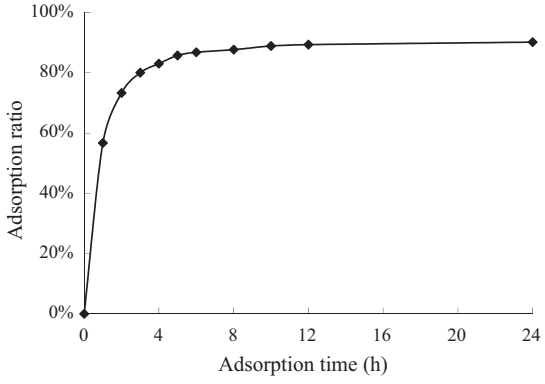

Fig. 1. Adsorption kinetics curve for gardenia yellow on HPD722 resin.

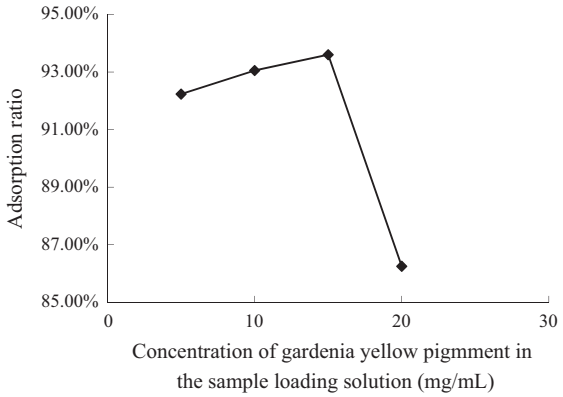

Fig. 2. Effect of concentration of gardenia yellow in the loading sample on the ratio of adsorption.

3.3. Optimization of the conditions for the purification of gardenia yellow

3.3.1. Effect of concentration of gardenia yellow in the loading sample on the ratio of adsorption

As shown in Fig. 2, the adsorption ratio increased slowly and then reduced as the concentration of gardenia yellow increased during the loading of the sample. Thus, the adsorption ratio was highest at a concentration of 15 mg/mL. When the concentration reached 20 mg/mL, the adsorption ratio logically reduced as the content of gardenia yellow in the loading sample exceed the adsorption saturation limit of the HPD722 resin. Furthermore, the use of a higher concentration of gardenia yellow during the loading of the sample would complicate the resin regeneration and recycling. So the appropriate concentration of gardenia yellow during the loading of the sample on the column to be 15 mg/mL. Thus, we calculated that the load capacity of HPD722 resin was 120 mg/g.

3.3.2. Effect of flow rate of loading sample on the ratio of adsorption

As shown in Fig. 3, it is evident that the sample flow rate had a direct effect on the ratio of the adsorption. Thus, with the increase of the sample flow rate, the adsorption ratio reduced gradually because of the apparent reduction in the time used for the adsorption with the increase of sample flow rate. The adsorption ratio was highest at a flow rate of 0.5 mL/min, however we concluded that the production period was too long and not conducive to industrial production. The adsorption ratio decreased only by 1.66% when the sample flow rate changed from 0.5 mL/min to 1.0 mL/min. Considering the production efficiency and the ratio of adsorption, the appropriate sample flow rate would be 1.0 mL/min.

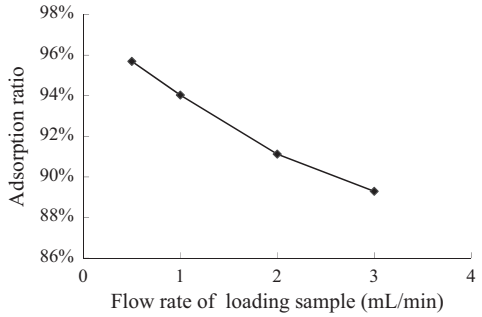

Fig. 3. Effect of flow rate of loading sample on the ratio of adsorption.

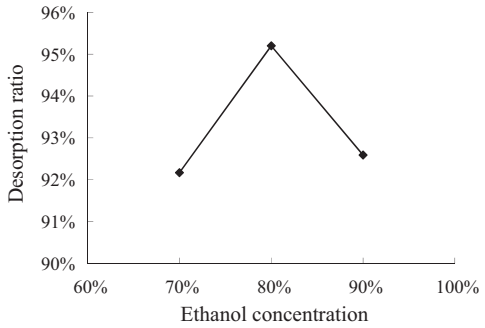

Fig. 4. Effect of ethanol concentration on the ratio of desorption.

3.3.3. Effect of ethanol concentration on the ratio of desorption

Ethanol–water solutions were usually used as the desorption solution of gardenia yellow [18,19]. Therefore, in this study we used different concentrations of ethanol–water solutions (70:30, 80:20, 90:10, v/v) to establish the proper desorption conditions. As shown in Fig. 4, the desorption ratio was highest when the ethanol concentration was 80%. Thus ethanol–water (80:20, v/v) solution was selected as the appropriate desorption solution.

3.3.4. Effect of desorption flow rate on the ratio of desorption

Desorption flow rate had obvious effect on the ratio of desorption. Too low desorption flow rate would result in long desorption time and more impurity desorbed. While too high desorption flow rate would increase the amount of desorption solution and result in heavy burden of subsequent enrichment process. From Fig. 5, it was seen that the ratio of desorption was highest at desorption flow rate of 3.5 mL/min.

3.3.5. Dynamic desorption curve of geniposide

The ethanol–water (15:85, v/v) solution was used to wash geniposide (the main impurity). The flow rate was 2.5 mL/min. As it is shown in Fig. 6, when the volume of ethanol–water (15:85, v/v) solution was at a range from 400 to 600 mL, the content of geniposide in the eluate was quite low and varied little. In order to remove geniposide as thoroughly as possible, the proper volume of ethanol–water (15:85, v/v) solution was 600 mL.

3.3.6. Dynamic desorption curve of gardenia yellow

According to previous results, the ethanol–water (80:20, v/v) solution was used to elute gardenia yellow and its flow rate was 3.5 mL/min. As can be seen in Fig. 7, the desorption curve was sharp, narrow, symmetric and with no trailing peak, which indicated that ethanol–water (80:20, v/v) solution was quite suitable as the desorption solution of gardenia yellow. When the consumption volume of eluent reached 200 mL, gardenia yellow was totally

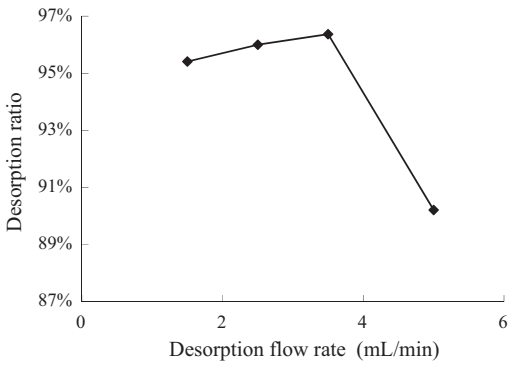

Fig. 5. Effect of desorption flow rate on the ratio of desorption.

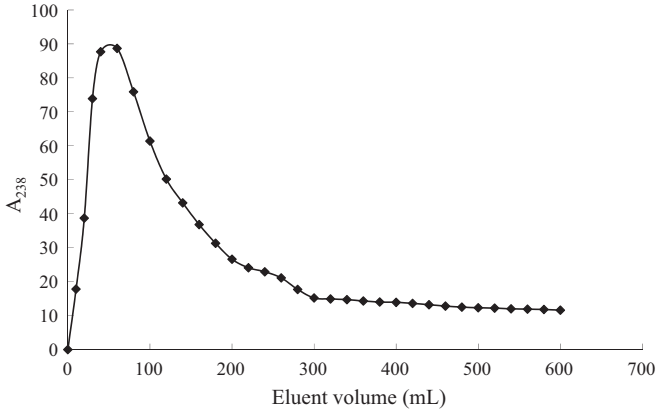

Fig. 6. Dynamic desorption curve of geniposide.

desorbed. Therefore, 200 mL was selected as the proper desorption volume.

3.4. UV–vis analysis of gardenia yellow before and after purification

The crude gardenia yellow was purified according to the above optimized refining process, and the eluate of ethanol–water (80:20, v/v) solution was collected and concentrated by rotary evaporation to remove the solvent, then dried thoroughly in a vacuum freeze-drier under −80 °C, finally the purified product was obtained. The result of color value analysis showed that colority of the product was 300. The UV–vis absorption spectrums of gardenia yellow solution before and after purification were shown in Figs. 8 and 9, respectively. There were three obvious absorption peeks (238 nm for geniposide, 323 nm for chlorogenic acid and 442 nm for gardenia yellow, respectively) in Fig. 8. By comparison, it can be seen that the absorption peek of geniposide almost disappeared and the absorption peek of chlorogenic acid was significantly smaller, while the relative peak of gardenia yellow increase obviously after the treatment with the HPD722 resin. The results indicated that most impurities such as geniposide and chlorogenic acid were removed.

3.5. HPLC analysis of crocin-1 and gardenia yellow after purification

Main components in gardenia yellow are crocin and crocetin. The HPLC chromatograms of crocin standard and gardenia yellow after purification were shown in Figs. 10 and 11, respectively. The results showed that the absorption peeks of gardenia yellow after

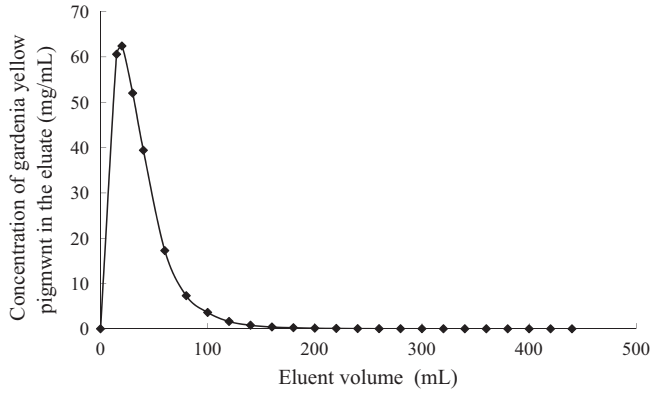

Fig. 7. Dynamic desorption curve of gardenia yellow.

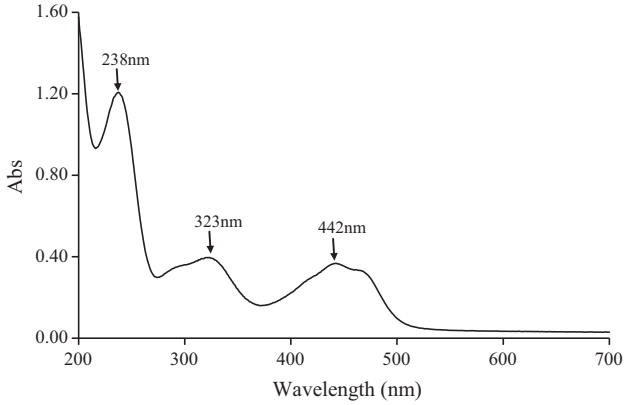

Fig. 8. Absorption spectrum of gardenia yellow solution before purification.

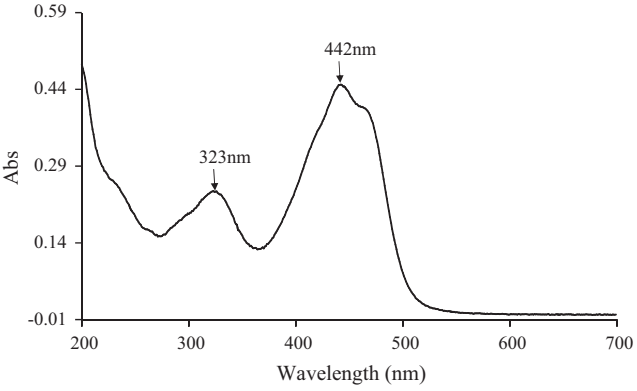

Fig. 9. Absorption spectrum of gardenia yellow solution after purification.

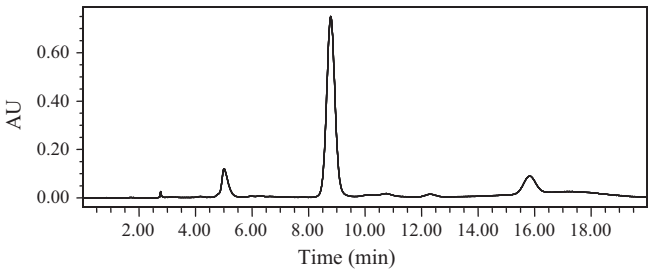

Fig. 10. HPLC spectrum of crocin-1.

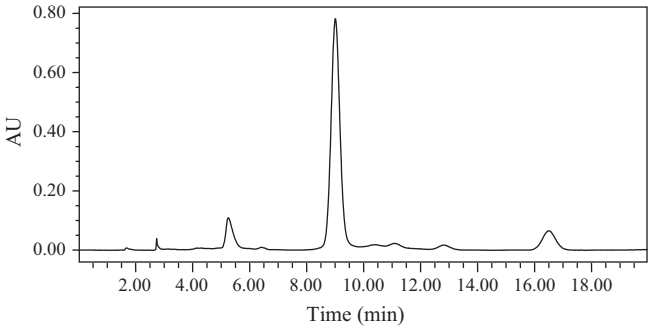

Fig. 11. HPLC spectrum of gardenia yellow solution after purification.

purification was similar with that of crocin-1 standard. It indicated that the purity of gardenia yellow after purification was high.

4. Conclusions

In this study, the adsorption and desorption characteristics of nine macroporous resins were investigated by static adsorption and desorption of gardenia yellow. HPD722 resin was selected to enrich and purify gardenia yellow because of its high adsorption capacity, high adsorption and desorption ratios as well as high selectivity. The processes of dynamic adsorption and desorption were conducted to ensure the optimal separation parameters of the HPD722 resin. Under the optimal separation parameters, the load capacity of HPD722 resin for gardenia yellow was 120 mg/g, which was 1.8 times of that of HPD450 resin [19]. It means that it would greatly reduce the amount needed of the selected resin and the cost. The colority of the gardenia yellow product obtained were 300. Furthermore, the results of UV–vis and HPLC analysis showed that most impurities were removed after purification with the HPD722 resin and the content of gardenia yellow in the product was almost the same as that (≥98%) of the standard.

In conclusion, this adsorption–desorption method was useful in the enrichment and purification of gardenia yellow from *G. jasminoides* var. *radicans Makino* for its low cost, high efficiency, procedural simplicity and ease in scaling-up.

References

[1] M.L. He, X.W. Cheng, J.K. Chen, T.S. Zhou, Chromatographia 64 (2006) 713.
[2] T. Watanabe, S. Terabe, J. Chromatogr. A 880 (2000) 311.
[3] J.W. Shao, J.F. Chen, Y.H. Guo, G.P. Li, L. Li, S.M. Peng, J.Z. Wu, J. Shenyang Agric. Univ. 37 (2006) 857.
[4] T.W. Chen, B.B. Kang, L.Y. Ren, S.J. Chen, H.T. Lin, Chin. J. Trop. Crops 31 (2010) 1398.
[5] F.X. Xie, J.M. Qiu, S.H. Tu, X.L. Xie, J. Nanchang Univ. Nat. Sci. 29 (2005) 278.
[6] A. Ozakia, M. Kitanob, N. Furusawac, H. Yamaguchic, K. Kurodaa, G. Endoa, Food Chem. Toxicol. 40 (2002) 1603.
[7] S. Pfister, P. Meyer, A. Steck, H. Pfander, J. Agric. Food Chem. 44 (1996) 2612.
[8] D.Q. Zhang, F.J. Lu, J.X. Tai, Q. Fu, Trans. CSAE 15 (1999) 226.
[9] X.L. Lu, Z.M. Yao, P.P. Jiang, Food Ferment. Ind. 27 (2001) 39.
[10] L.Y. Ren, Master Fujian Agric. Univ. (2009).
[11] C.P. Wang, J.B. Ji, L.H. Wang, T.H. Bao, Chem. Ind. Eng. Prog. 22 (2003) 622.
[12] F.X. Xie, Z.M. Zu, S.H. Sheng, X.L. Xie, J. Nanchang Univ. Eng. Technol. 27 (2005) 49.
[13] M. Gao, W. Huang, C.Z. Liu, J. Chromatogr. B 858 (2007) 22.
[14] G.T. Jia, X.Y. Lu, J. Chromatogr. A 1193 (2008) 136.
[15] Z.F. Hou, G. Wang, G.X. Sun, Lishizhen Med. Mater. Med. Res. 21 (2010) 2943.
[16] GB7912-2010, CNS.
[17] Q. Duan, S.H. Chen, Tradit. Chin. Drug Res. Pharmacol. 21 (2010) 299.
[18] Z.S. Zhong, J.X. Pan, Y.K. Chen, Fine Chem. 24 (2007) 581.
[19] J.W. Hu, W. Xiong, X.H. Li, Q.R. Ji, Z.X. Tu, Nat. Prod. Res. Dev. 23 (2011) 304.