

Article

Adsorption and Desorption Characteristics of Simiao Yong'an Decoction on Macroporous Adsorption Resins

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Abstract: In this study, to identify an appropriate macroporous resin for the purification of the crude extract derived from Simiao Yong'an Decoction (SMYAD), five distinct resins were evaluated based on their adsorption and desorption performance. Through this comparative analysis, D101 macroporous adsorption resin was determined to be the most suitable candidate. Investigations into the adsorption mechanism revealed that the process followed pseudo-second-order kinetics and was well described by the Langmuir isotherm model. Thermodynamic analysis further indicated the spontaneous and exothermic nature of the adsorption process. Through systematic optimization, the ideal purification parameters for SMYAD were established as follows: sample loading volume—6 bed volumes (BV), eluent concentration—90% ethanol (v/v), and eluent volume—3 BV, with both adsorption and desorption flow rates maintained at 3 BV/h. Finally, the purification effect was evaluated by fingerprint and similarity analysis. Under the optimal purification process, the content of the five index components was as follows: harpagide (4.26 mg/g), chlorogenic acid (38.37 mg/g), ferulic acid (4.78 mg/g), liquiritin (7.42 mg/g), and harpagoside (10.25 mg/g). The yield of the five components ranged from 70.74% to 86.26%. In conclusion, this approach demonstrates rapid processing capabilities and high efficiency, offering valuable methodological insights for the isolation and purification of complex traditional Chinese medicine formulations using macroporous adsorption resins.



Academic Editor: Xinhua Qi

Received: 21 February 2025

Revised: 28 March 2025

Accepted: 30 March 2025

Published: 31 March 2025

Citation: Jiao, W.; Liu, E.; Luo, Y.; Niu, Y.; Guo, Y. Adsorption and Desorption Characteristics of Simiao Yong'an Decoction on Macroporous Adsorption Resins. *Separations* **2025**, *12*, 84. <https://doi.org/10.3390/separations12040084>

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Keywords: Simiao Yong'an decoction; Chinese herbal medicine; macroporous resins; adsorption kinetics; adsorption thermodynamics

1. Introduction

Simiao Yong'an Decoction (SMYAD) comes from the second volume of the New Compilation of Yan Fang. It is composed of honeysuckle, scrophularia, angelica, and licorice at a ratio of 3:3:2:1. It has the functions of clearing heat and detoxing, activating blood circulation and removing blood stasis [1–3]. At present, it is widely used in coronary atherosclerosis [4–7], diabetic foot and diabetic macroangiopathy [8–10], gout, and other aspects [11,12]. The study of chemical composition showed that SMYAD contained flavonoids, saponins, iridoids, organic acids, and other types of compounds [13].

The therapeutic effectiveness of Traditional Chinese Medicine (TCM) stems from the synergistic interactions among multiple bioactive constituents within herbal formulations. Consequently, conventional quality assessment methods focusing on individual components prove inadequate for comprehensive evaluation of TCM preparations, and it

is necessary to establish control methods and means that can reflect the overall quality of TCM. According to the current scientific development and technical level, the integration of chromatographic fingerprint analysis with multi-constituent quantification and specific marker identification represents a viable strategy for the holistic quality assessment of Traditional Chinese Medicine, aligning with the fundamental principles of TCM's holistic approach [14–17].

Macroporous adsorption resin technology is a technology that uses the porous structure and selective adsorption function of macroporous adsorption resin to separate refined effective components from TCM extract [18,19]. It is an organic polymer that is insoluble in acids, bases, and various organic solvents. Its pore size and specific surface area are relatively large. It has a three-dimensional pore structure inside the resin, and its adsorption capacity is large, selective, and fast [20,21]. In recent years, this method has been widely used for the isolation and purification of bioactive compounds from natural sources [22–25]. At present, macroporous resins are mostly used to separate and purify single components or similar components, and there are few studies on the separation and purification of multiple components of traditional Chinese medicine compounds. The chemical composition of TCM decoction is affected by the process of decocting and the interaction between components. The measurement and control of a single component drug can not fully represent the overall quality of the decoction, nor can it scientifically reflect the integrity of its curative effect. Therefore, it is necessary to analyze and control the chemical composition of the whole decoction, and the macroporous adsorption resin can not only remove some impurities and improve the purity of the active ingredient but also ensure the stability and uniformity of the traditional Chinese medicine preparation.

This research established an integrated protocol for SMYAD preparation involving aqueous-phase extraction coupled with macroporous resin purification. Parameter optimization was conducted to examine critical variables affecting adsorption–desorption dynamics, complemented by mechanistic interpretation of resin–analyte interactions. Chromatographic fingerprint profiling was implemented as a quality control metric to validate purification efficiency. The optimized methodology not only facilitates standardized production of SMYAD formulations but also advances theoretical understanding of macroporous resin applications in phytopharmaceutical processing, thereby contributing methodological insights for traditional medicine modernization initiatives.

2. Materials and Methods

2.1. Materials and Reagents

The experimental study utilized five distinct macroporous adsorption resins, commercially sourced from Tianjin Damao Chemical Reagent Factory (Tianjin, China). The pretreatment process of the macroporous adsorption resin consisted of three steps: first, the surface was rinsed with 95% (*v/v*) ethanol, followed by 24 h impregnation at room temperature, and finally, the solvent replacement was completed by repeated rinsing with distilled water. The adsorption–desorption efficiency of compounds is critically influenced by resin polarity coupled with key physical characteristics, particularly surface area dimensions, pore size distribution, and moisture retention capacity [24,26]. The investigation employed various resin types categorized by their polarity characteristics: non-polar (D101, X-5), weakly polar (AB-8), and polar (NKA-9, S-8), each exhibiting distinct physical properties, as detailed in Table 1. All resin information comes from the resin manufacturer. Phosphoric acid (H_3PO_4 , HPLC grade) was purchased from Tianjin Kemio Chemical Reagent Ltd. (Tianjin, China). Acetonitrile (ACN, HPLC grade) was obtained from Concord Technology Ltd. (Tianjin, China). The chemical reference standards and SMYAD compound authentication materials were commercially acquired from Chengdu Ruifensiedan Biotechnology Co.,

Ltd. (Chengdu, China). The four herbal materials—honeysuckle, scrophularia, angelica, and licorice—were sourced from their respective primary cultivation regions.

Table 1. Physicochemical properties on NKA-9, S-8, AB-8, X-5, D101.

Resin	Polarity	Diameter (mm)	Surface Area (m ² /g)	Average Pore Size (Å)
NKA-9	Polar	0.3–1.25	250–290	155–165
S-8	Polar	0.3–1.25	100–120	280–300
AB-8	Weak-polar	0.3–1.25	480–520	130–140
X-5	Non-polar	0.3–1.25	500–600	290–300
D101	Non-polar	0.3–1.25	550–600	90–110

2.2. Sample Preparation

First, 112.5 g of honeysuckle, 112.5 g of scrophularia, 75 g of angelica, and 37.5 g of licorice were weighed. Following the addition of 3375 mL of purified water, the mixture underwent a 30 min soaking period, succeeded by a two-stage decoction process: an initial 2 h extraction followed by a subsequent 1 h treatment. Then it was heated to a boil and kept slightly boiling. After decoction, the liquid medicine was combined and left to cool. After cooling, filter papers with diameters of 10 µm, 1 µm, and 0.45 µm were used for press filtration in order.

2.3. HPLC Analysis of SMYAD

The chromatographic characterization of SMYAD was conducted using a Hitachi PM1000 HPLC platform (Hitachi Limited, Tokyo, Japan) equipped with a quaternary solvent delivery system, automated sample injector, and ultraviolet detection unit. Compound separation was accomplished on a thermostatically controlled (38 °C) Kromasil C18 analytical column (250 × 4.6 mm, 5 µm particle size). The elution process employed a binary solvent system consisting of acetonitrile (mobile phase A) and 0.4% phosphoric acid aqueous solution (*v/v*, mobile phase B), maintained at a constant flow rate of 1 mL/min with programmed gradient conditions (Table 2). Detection parameters included 210 nm wavelength and 10 µL injection volume. As shown in Figure 1, reference compounds exhibited distinct retention times: harpagide (7.887 min), chlorogenic acid (17.847 min), ferulic acid (33.560 min), liquiritin (35.360 min), and harpagoside (56.900 min). External standard calibration curves demonstrated excellent linearity ($R^2 \geq 0.9996$) across 0.0016–1.46 mg/mL, with regression equations as follows: $y_{chl} = 1 \times 10^7x + 202,131$ ($R^2 = 0.9997$), $y_{fer} = 5 \times 10^7x - 82,958$ ($R^2 = 0.9998$), $y_{liq} = 6 \times 10^6x - 31,397$ ($R^2 = 0.9999$), $y_{hgi} = 2 \times 10^6x - 9692$ ($R^2 = 0.9999$), and $y_{hgo} = 1 \times 10^7x - 31,276$ ($R^2 = 0.9996$). In the established calibration model, *y* represents the chromatographic peak area of the target compound, while *x* denotes the corresponding concentration (mg/mL) for each of the five analytes.

Table 2. HPLC gradient elution method for SMYAD: acetonitrile (A) and 0.4% (*v/v*) phosphoric acid (mobile phase B).

Time (Min)	A (%)	B (%)
0	5	95
3	5	95
18	11	89
45	20	80
55	33	67
60	50	50
70	100	0

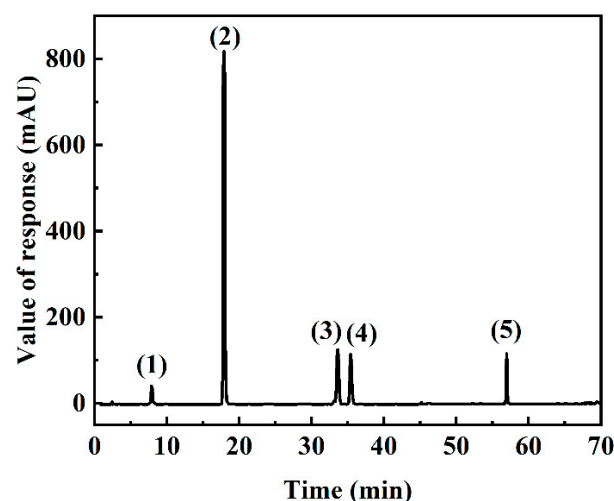


Figure 1. HPLC chromatograms of standards: harpagide (1), chlorogenic acid (2), ferulic acid (3), liquiritin (4), and harpagoside (5).

2.4. Static Screening of Resins

Following preconditioning, precisely weighed 2.0 g aliquots of macroporous resins were introduced into individual conical flasks, each containing 50 mL of SMYAD solution. The flasks were subsequently subjected to continuous shaking at 120 rpm in a temperature-controlled incubator shaker maintained at 25 °C for a duration of 24 h. Aliquots (0.5 mL) collected pre- and post-adsorption were membrane-filtered (0.22 µm) for HPLC analysis. Resin particulates were isolated via filtration, rinsed with ultrapure water, and then subjected to ethanol desorption (70% *v/v*, 50 mL) under identical agitation parameters (120 rpm, 25 °C, 24 h).

The quantitative evaluation of adsorption performance was determined through two key parameters: adsorption capacity (q_t , mg/g) and adsorption rate (%), which were mathematically derived using Equations (1) and (2), respectively.

$$q_t = \frac{(C_0 - C_t)V_1}{m} \quad (1)$$

$$A = \frac{(C_0 - C_t)}{C_0} \times 100\% \quad (2)$$

In these equations, q_t represents the adsorption capacity at specific time point t (mg/g), while A denotes the adsorption efficiency (%). The parameters are defined as follows: C_0 corresponds to the initial concentration in the SMYAD crude extract (mg/mL), C_t indicates the residual concentration in the supernatant after adsorption (mg/mL), V_1 signifies the total volume of the extraction solution (mL), and m refers to the mass of the dehydrated resin (g).

The evaluation of desorption performance was quantified through two principal metrics: desorption capacity (q_d , mg/g) and desorption ratio (D , %), which were computationally determined using Equations (3) and (4).

$$q_d = \frac{C_1 V_2}{m} \quad (3)$$

$$D = \frac{C_1 V_2}{(C_0 - C_t)V_1} \times 100\% \quad (4)$$

In the desorption equations, q_d signifies the desorbed quantity per unit mass (mg/g), while D represents the desorption yield (%). The parameters are defined as follows: C_1 is the

equilibrium concentration in the desorption solution (mg/mL), and V_2 is the total eluent volume (mL), with C_0 , C_t , m , and V_1 maintaining their previously established definitions from the adsorption parameters.

2.5. Adsorption Kinetics on D101 Resin

First, 50 mL of SMYAD solution was added to a stopped conical flask to mix with the pretreated resin (2.0 g). Then, the flask was shaken continuously with a constant speed (120 rpm) at 25 °C for 24 h. During the shaking process, aliquots (0.5 mL) were periodically collected for analysis at predetermined intervals spanning 0–24 h. Subsequently, kinetic data before adsorption equilibrium were selected and analyzed using two traditional adsorption models, which are expressed as follows:

Pseudo-first-order model equation:

$$\ln(q_e - q_t) = \ln q_e - K_1 t \quad (5)$$

Pseudo-second-order model equation:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \quad (6)$$

In these kinetic models, K_1 and K_2 denote the rate constants for adsorption processes, while q_e and q_t correspond to the equilibrium adsorption capacity and the capacity at time t , respectively [27–30].

2.6. Adsorption Isotherms and Heat of Adsorption

To characterize the adsorption behavior of SMYAD on D101, the effects of temperature (25, 35, and 45 °C) on adsorption were evaluated. The SMYAD solution was diluted into 10 parts by gradient and adsorbed by D101 resin. The SMYAD solutions were composed of harpagide (0.1025, 0.205, 0.3075, 0.41, 0.5125, 0.615, 0.7175, 0.82, 0.9225, and 1.025 mg/mL), chlorogenic acid (0.3162, 0.6324, 0.9486, 1.2648, 1.581, 1.8972, 2.2134, 2.5296, 2.8458, and 3.162 mg/mL), ferulic acid (0.2204, 0.4408, 0.6612, 0.8816, 1.102, 1.3224, 1.5428, 1.7632, 1.9836, and 2.204 mg/mL), liquiritin (0.2262, 0.4524, 0.6786, 0.9048, 1.131, 1.3572, 1.5834, 1.8096, 2.0358, and 2.262 mg/mL) and harpagoside (0.1073, 0.2146, 0.3219, 0.4292, 0.5365, 0.6438, 0.7511, 0.8584, 0.9657, and 1.073 mg/mL). Finally, 50 mL of SMYAD solution was added to a stopped conical flask to mix with the pre-treated resin (2.0 g). Then, the flask was shaken continuously with a constant speed (120 rpm) at 25 °C, 35 °C, or 45 °C for 24 h. The equilibrium concentrations of each component at different temperatures were determined by HPLC. The Langmuir and Freundlich isotherm models represent two widely utilized theoretical frameworks for characterizing adsorption phenomena in various materials.

The Langmuir isotherm can be mathematically formulated as follows:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (7)$$

The Freundlich adsorption isotherm can be mathematically formulated as follows:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (8)$$

In this context, q_m represents the theoretical maximum adsorption capacity (mg/g), while C_e indicates the equilibrium concentration in solution (mg/mL). The model parameters include K_L (Langmuir equilibrium constant), K_F (Freundlich capacity coefficient), and $1/n$ (heterogeneity factor characterizing adsorption site energy distribution) [31–35].

The thermodynamic analysis was performed by relating the K_L value of the Langmuir isotherm to the heat of adsorption. This energy is calculated using established thermodynamic formulas such as:

$$\ln K_L = \ln K_{L0} + \frac{Q}{RT} \quad (9)$$

In the thermodynamic analysis, K_{L0} represents the equilibrium constant at the reference temperature, and Q represents the heat of adsorption (kJ/mol), with R corresponding to the universal gas constant (8.314 J/mol·K) and T indicating the absolute temperature in Kelvin [26,35].

2.7. Optimization of Dynamic Adsorption and Desorption for D101 Resin

This study systematically investigated four key parameters affecting SMYAD purification: sample loading volume, ethanol elution gradient, elution volume, and flow rate. The dynamic adsorption capacity was evaluated by delivering SMYAD solutions (1–10 BV) through a D101 resin column (300 mm × 50 mm id) at 3.0 BV/h, with 10 mL effluent collected per 120 mL BV for HPLC analysis. Following resin saturation, gradient elution experiments were performed using sequential 3 BV volumes of water and ethanol solutions (10–90% *v/v*) to determine optimal ethanol concentration. Subsequent investigations examined elution volume effects by employing 3 BV water followed by 1–10 BV of 90% ethanol. Finally, flow rate optimization was conducted using 3 BV water and 90% ethanol at rates ranging from 1.0 to 5.0 BV/h. All collected fractions were quantitatively analyzed by HPLC to determine component purity and recovery rates.

2.8. Determination of Elution Product by HPLC

Chromatographic fingerprints of SMYAD crude/purified extracts were acquired using HPLC and subsequently subjected to similarity analysis (TCMC-FPSES 2012 software). The system quantified pre- vs. post-purification chromatogram congruency through built-in algorithms. Following the extraction protocol outlined in Section 2.2, crude SMYAD containing four target compounds was prepared. Post-quantification, the extract was bifurcated: one aliquot underwent lyophilization for mass determination, while the counterpart was processed through the optimized purification workflow. Comparative HPLC profiling of both fractions enabled component purity calculation via Equation (10), revealing purification-induced purity modulation.

$$p = \frac{C_i V}{m} \times 100\% \quad (10)$$

In the purity calculation formula, p denotes the percentage purity, where C_i corresponds to the concentration of individual SMYAD components (mg/mL), V represents the total sample volume (mL), and m indicates the mass of the lyophilized product (g).

2.9. Statistical Analysis

Statistical analysis and data organization were performed using Microsoft Excel 2021, with experimental results expressed as mean ± standard deviation from triplicate determinations. Graphical representations were generated using Origin 2021 software (Origin Lab Corporation, Northampton, MA, USA). Chromatographic profile analysis was conducted according to Traditional Chinese Medicine Chromatographic Finger-print Similarity Evaluation System 2012 (Chinese Pharmacopoeia Commission, Beijing, China).

3. Results and Discussion

3.1. Static Adsorption and Desorption

Through comprehensive evaluation of adsorption–desorption characteristics across five macroporous resins (Figure 2), D101 demonstrated superior performance, exhibiting the highest adsorption capacity and significantly enhanced desorption efficiency for SMYAD components. This non-polar resin was consequently chosen for subsequent purification processes, as its physicochemical properties align with the principle of “like dissolves like,” facilitating effective adsorption and desorption of non-polar compounds such as liquiritin and harpagoside. Furthermore, resin performance is influenced by structural parameters, including specific surface area and pore diameter distribution, which directly affect adsorption capabilities [26]. Comparative analysis revealed that D101 possessed the most extensive specific surface area among the evaluated resins, suggesting that increased surface area enhances the interaction and mass transfer of polarity-compatible compounds during adsorption–desorption processes.

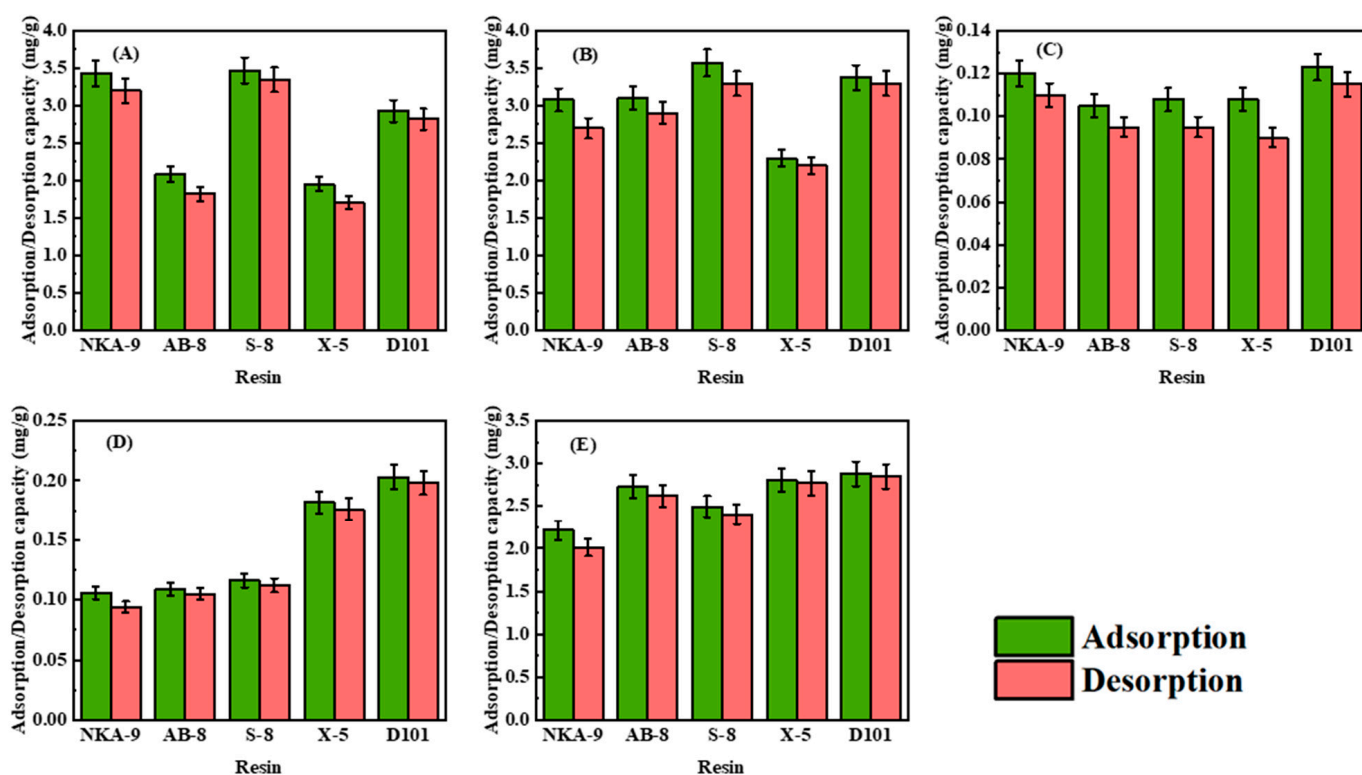


Figure 2. Static adsorption–desorption of harpagide (A), chlorogenic acid (B), ferulic acid (C), liquiritin (D), and harpagoside (E) on five resins.

Figure 3A illustrates the temporal adsorption profile of D101 resin under static conditions. All five components exhibited similar adsorption kinetics, characterized by rapid initial uptake within the first hour, followed by a gradual decline in adsorption rate over 1–2 h, ultimately reaching equilibrium at 6 h. Based on these observations, 6 h was established as the optimal adsorption duration. Corresponding desorption kinetics (Figure 3B) demonstrated peak efficiency within 1 h, establishing this timeframe as optimal for desorption.

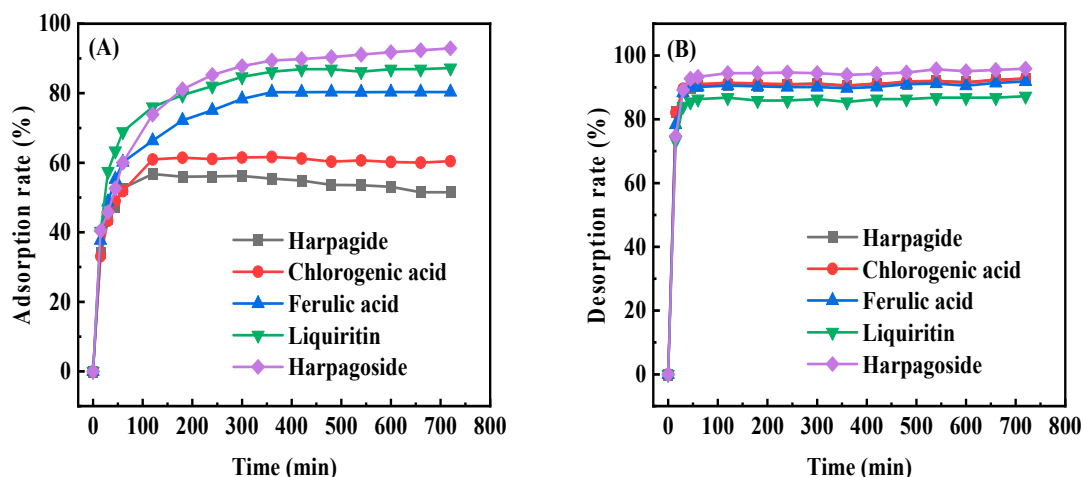


Figure 3. Results of static adsorption (A) and desorption (B) equilibrium time investigation.

3.2. Adsorption Kinetics of SMYAD on D101 Resin

The kinetic analysis, presented in Figure 4 and Table 3, revealed that the pseudo-second-order (PSO) model provided superior fitting for describing SMYAD adsorption on D101 resin. The close agreement between theoretical q_e values from the PSO model and experimental data further validated this observation. Therefore, the quasi-second-order kinetic model is more beneficial to describe the adsorption mechanism during the purification of SMYAD.

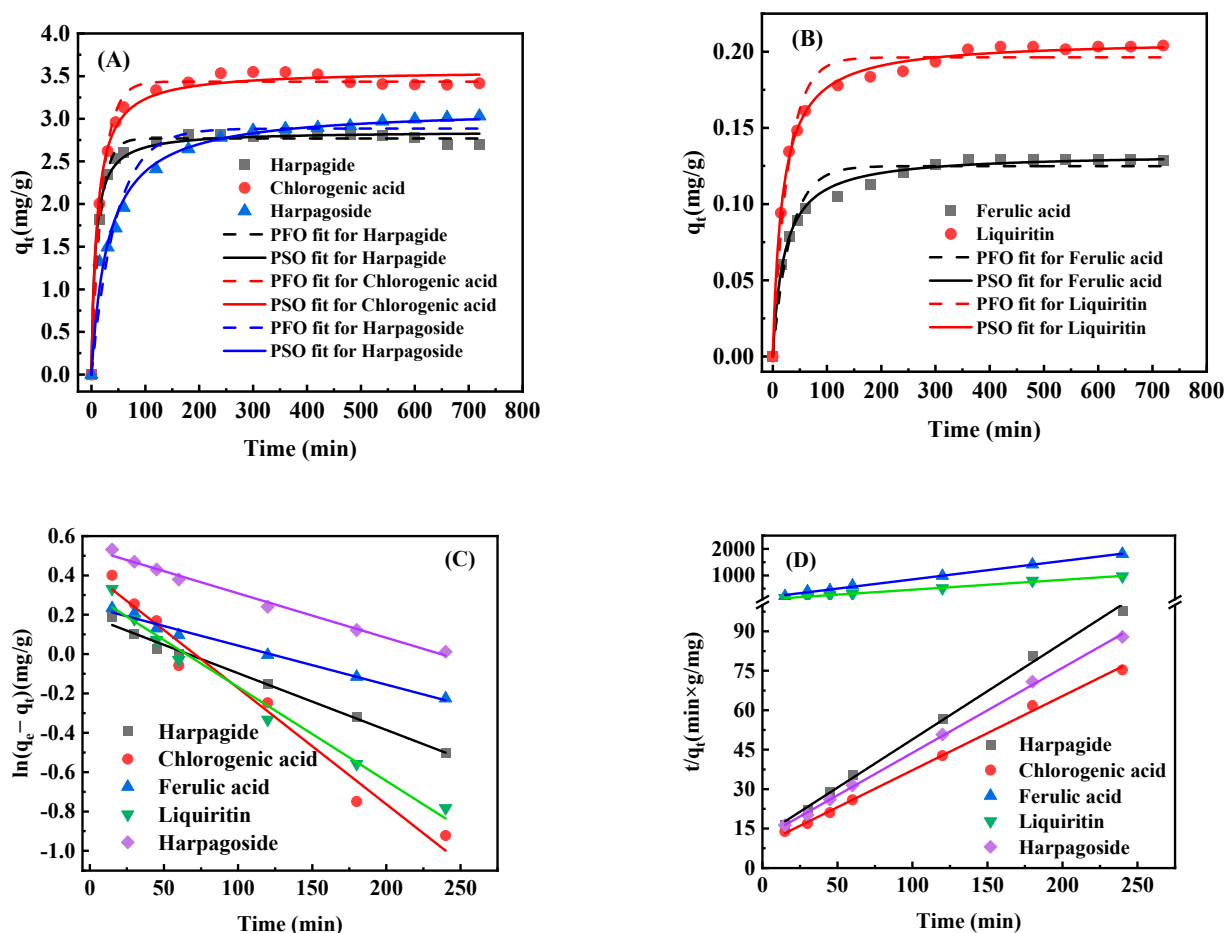


Figure 4. Adsorption kinetic curves of SMYAD on the D101 resin, (A,B). The linear correlations based on PFO (C) and PSO (D).

Table 3. Kinetic model fitting equations and model parameters.

Components	C_0 (mg/mL)	$q_{e,exp}$ (mg/g)	PFO Parameters			PSO Parameters		
			K_1 (min^{-1})	$q_{e,1}$ (mg/g)	R^2	K_2 ($\frac{\text{g}}{\text{mg} \times \text{min}}$)	$q_{e,2}$ (mg/g)	R^2
Harpagide	0.198	2.702	0.00288	1.211	0.989	0.0110	2.726	0.997
Chlorogenic acid	0.943	3.416	0.00590	1.518	0.969	0.00881	3.548	0.998
Ferulic acid	0.234	0.129	0.00199	1.274	0.983	0.308	0.144	0.996
Liquiritin	0.117	0.204	0.00477	1.363	0.976	0.157	0.268	0.995
Harpagoside	0.202	3.031	0.00226	1.707	0.987	0.00906	3.096	0.999

3.3. Adsorption Isotherms and Heat of Adsorption on D101 Resin

Experimental data analysis yielded the Langmuir and Freundlich isotherm parameters presented in Table 4. The Langmuir model demonstrated superior fitting for all five components' adsorption on D101 resin, indicating monolayer adsorption at the solid–liquid interface [36,37]. Figure 5 illustrates the temperature-dependent adsorption isotherms, revealing optimal adsorption efficiency at 25 °C.

Table 4. Isothermal parameters of SMYAD on D101 resin.

Components	Temperature (°C)	Langmuir Equation			Freundlich Equation		
		K_L (mL/mg)	q_m (mg/g)	R^2	$K_F[(\text{mg/g})^{1/n}]$ (mL/mg) $^{1/n}$	$1/n$	R^2
Harpagide	25	40.75	5.45	0.98	5.92	0.15	0.89
	35	50.48	4.61	0.99	4.90	0.14	0.88
	45	64.58	4.03	0.98	4.11	0.10	0.87
Chlorogenic acid	25	5.44	6.28	0.97	5.04	0.18	0.83
	35	7.83	5.45	0.99	4.63	0.14	0.84
	45	9.94	5.09	0.99	4.43	0.12	0.87
Ferulic acid	25	2.21	5.53	0.97	3.62	0.38	0.89
	35	3.13	4.19	0.94	3.03	0.31	0.83
	45	3.72	3.55	0.97	2.55	0.31	0.89
Liquiritin	25	2.54	4.42	0.96	3.02	0.34	0.86
	35	3.23	3.49	0.94	2.54	0.29	0.81
	45	3.70	2.88	0.96	2.17	0.26	0.84
Harpagoside	25	12.70	9.91	0.99	10.99	0.25	0.90
	35	16.48	8.60	0.99	9.27	0.28	0.89
	45	22.72	7.43	0.97	7.67	0.28	0.85

Thermodynamic analysis utilizing the Langmuir equilibrium constant (K_L) revealed exothermic adsorption characteristics for all five components, as depicted in Figure 6. The linear regression coefficients (R^2) were determined as follows: harpagide (0.9485), chlorogenic acid (0.9999), ferulic acid (1), liquiritin (0.9990), and harpagoside (0.9385). Calculated adsorption heats (Q) derived from Equation (9) were −12.2274, −15.2479, −14.4414, −10.3567, and −15.3451 kJ/mol for respective components. Q is less than 0, indicating exothermic adsorption; all below the 43 kJ/mol threshold indicate physical adsorption processes [24,26].

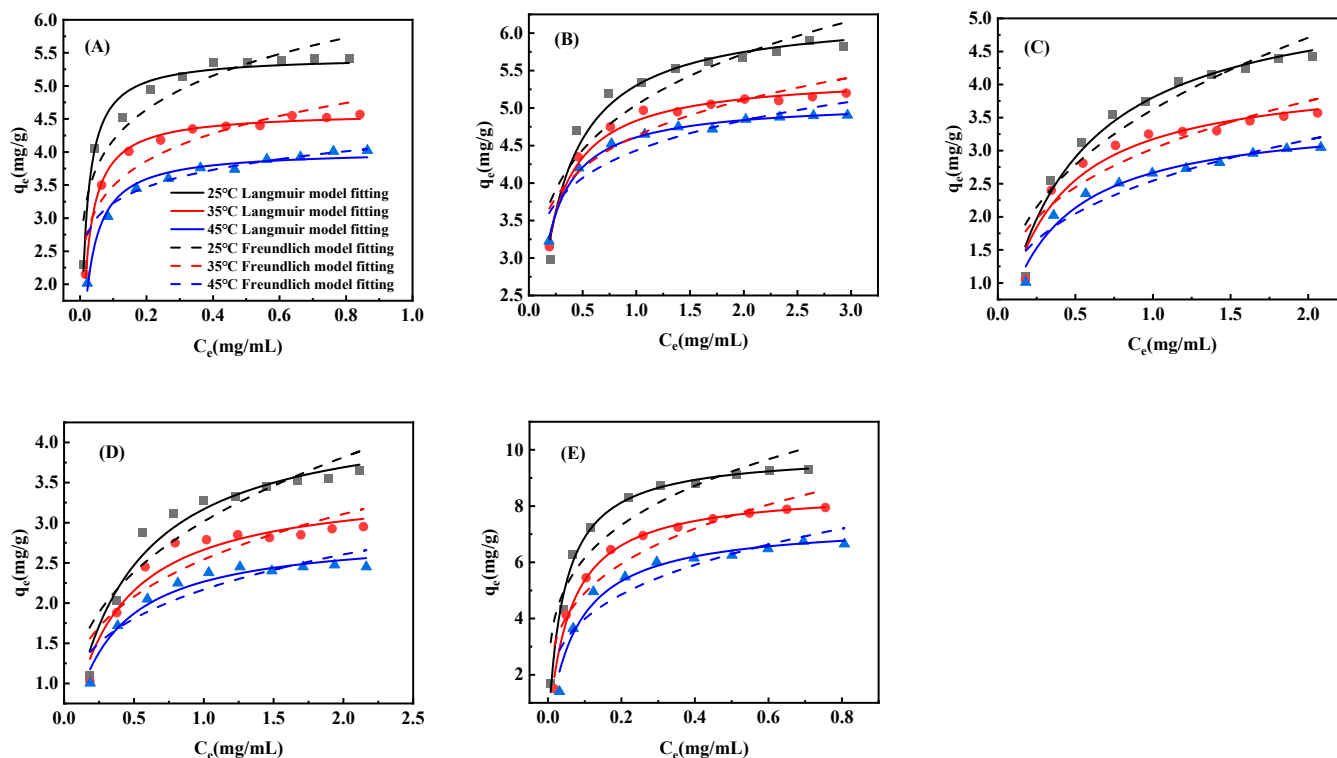


Figure 5. Results of Langmuir model and Freundlich model fit for harpagide (A), chlorogenic acid (B), ferulic acid (C), liquiritin (D), and harpagoside (E) at 25, 35, and 45 °C.

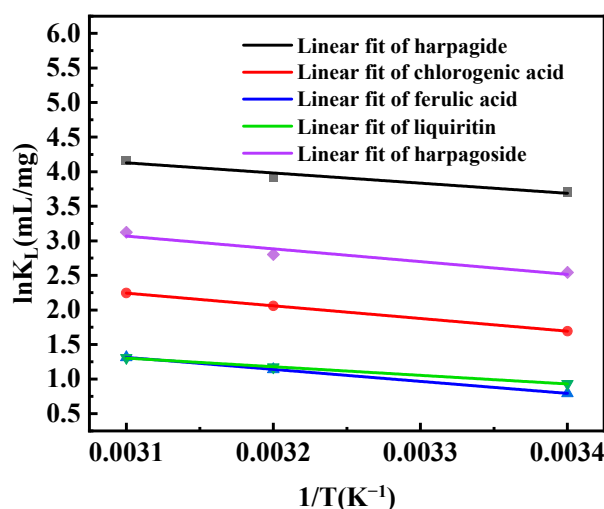


Figure 6. Results of linear fit between $\ln K_L$ and $1/T$.

3.4. Dynamic Adsorption and Desorption of SMYAD on D101 Resin

Figure 7A illustrates that chlorogenic acid, liquiritin, and harpagide exhibited initial leakage, whereas ferulic acid and harpagoside remained retained. The leakage point is operationally defined as the effluent index component concentration reaching 10% of its corresponding loading solution concentration. At 6 BV outflow volume, chlorogenic acid concentration in the effluent measured 0.07 mg/mL, approximating 10% of its loading solution concentration (0.7 mg/mL), thereby constituting the leakage threshold. Concurrently, the remaining four index components demonstrated marked leakage. Consequently, 6 BV was empirically established as the optimal loading volume for D101 macroporous resin.

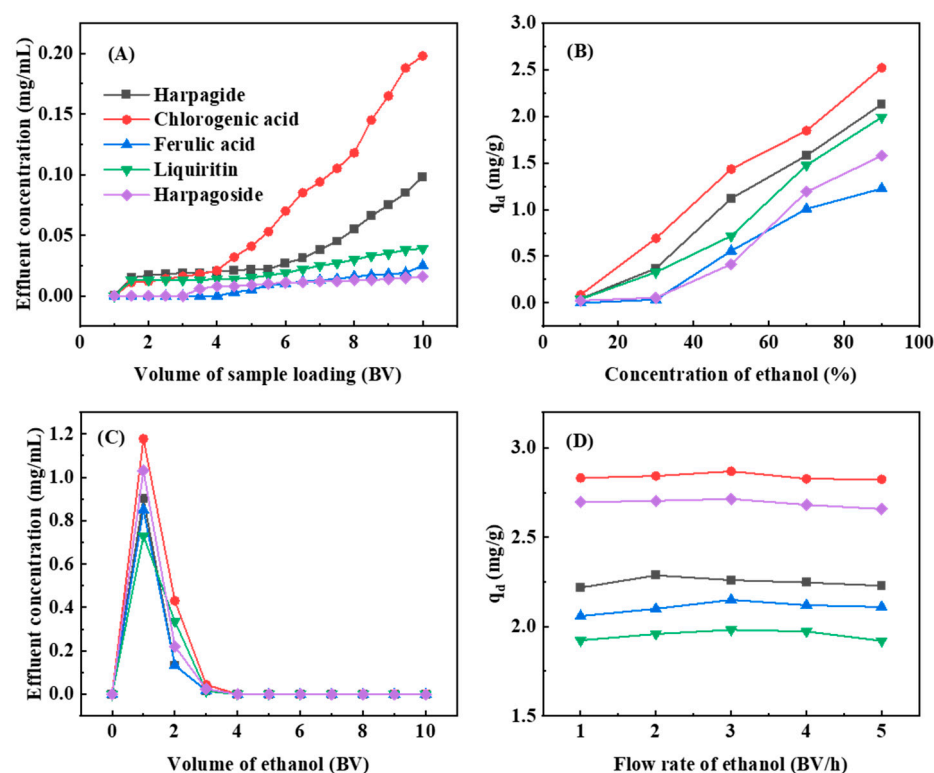


Figure 7. Dynamic adsorption–desorption results of SMYAD on D101 resin: sample loading volume (A), concentration of eluate (B), volume of eluate (C), and flow rate of eluate (D).

Water wash volume was optimized to 3 BV using Molish reaction monitoring. Figure 7B demonstrates ethanol concentration-dependent desorption behavior of the five target components, with desorption yields progressively rising as ethanol concentration increased. Consequently, 90% (*v/v*) ethanol was validated as the optimal eluent for subsequent purification steps.

The dynamic desorption curves were determined by HPLC, and the results are shown in Figure 7C: effluent concentrations initially increased with eluate volume, peaking at 1 BV before subsequent decline. The five target components exhibited progressive concentration reduction beyond 2 BV, culminating in complete elution at 3 BV.

As illustrated in Figure 7D, optimal desorption efficiency was achieved at 3 BV/h for chlorogenic acid, ferulic acid, liquiritin, and harpagoside, with harpagide showing slightly reduced performance. This flow rate was subsequently identified as the optimal parameter, balancing desorption yield with operational efficiency.

3.5. Evaluation of Purification Effect of D101 Resin

Systematic optimization of macroporous resin parameters demonstrated >70% retention efficiency for active constituents and 91.81% solid mass reduction, with both parameters exhibiting <2% RSD, confirming process stability. Post-purification analysis revealed a substantial decrease in extract mass per prescription unit from 15.072 g to 1.2339 g. The dried extract contained quantified concentrations of harpagide (4.26 mg/g), chlorogenic acid (38.37 mg/g), ferulic acid (4.78 mg/g), liquiritin (7.42 mg/g), and harpagoside (10.25 mg/g), achieving enhanced component enrichment despite reduced total extract yield. Comprehensive retention and purity metrics are tabulated in Table 5.

Table 5. The results of purity and solids reduction rate.

	Purity (%)			Reserved Rate (%)	RSD
	Before	After	RSD		
Harpagide	0.82	4.94	0.04	80.32	0.02
		5.31		82.71	
		5.42		83.78	
Chlorogenic acid	2.48	10.98	0.05	81.33	0.03
		11.30		82.72	
		11.73		83.77	
Ferulic acid	0.76	3.82	0.06	70.74	0.03
		4.07		71.43	
		4.77		79.62	
Liquiritin	1.03	6.94	0.03	76.33	0.05
		7.25		85.13	
		7.31		86.26	
Harpagoside	1.17	7.95	0.03	78.73	0.02
		8.03		79.32	
		8.12		80.61	

The chromatographic profiles of crude and purified SMYAD extracts were evaluated using the 2012 edition of the Traditional Chinese Medicine Chromatographic Fingerprint Similarity Evaluation System (TCMCFSES). Using the crude extract chromatogram (S1) as the reference profile, median-based peak alignment identified 26 consensus peaks across samples, with five authenticated as harpagide, chlorogenic acid, ferulic acid, liquiritin, and harpagoside (Figure 8). Quantitative similarity analysis (Table 6) revealed inter-batch similarity indices exceeding 0.998. The results showed that the active components were well preserved before and after purification, and the purification process of SMYAD by D101 macroporous adsorption resin was stable with good repeatability.

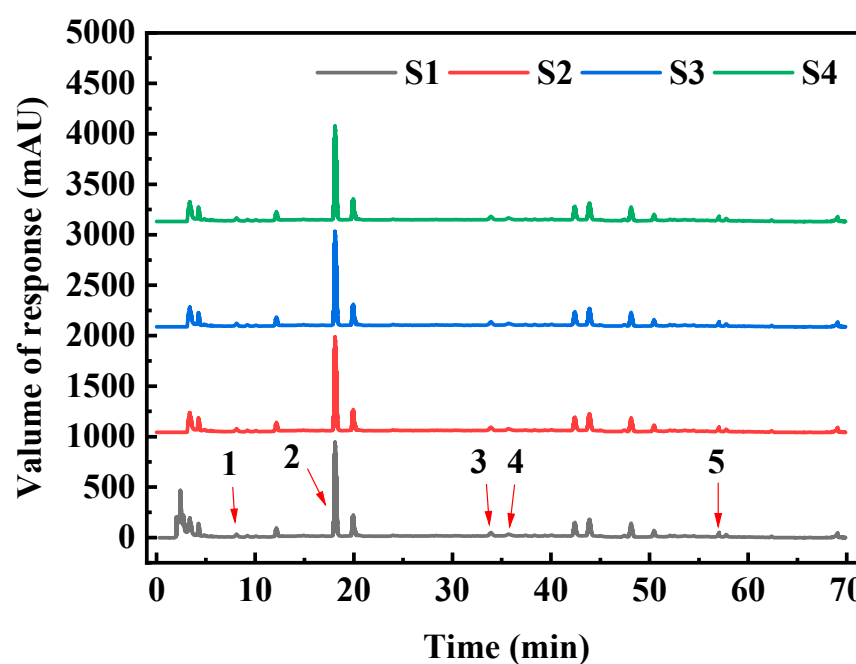


Figure 8. SMYAD fingerprint chromatogram. 1-Harpagide, 2-Chlorogenic acid, 3-Ferulic acid, 4-Liquiritin, and 5-Harpagoside. S1 represents the crude SMYAD extract, while S2–S4 denote the purified SMYAD.

Table 6. Results of similarity evaluation.

	S1	S2	S3	S4
S1	1	0.999	0.999	0.999
S2	0.999	1	0.998	0.999
S3	0.999	0.998	1	0.998
S4	0.999	0.999	0.998	1

4. Conclusions

D101 macroporous resin was identified as optimal among five candidate resins for SMYAD crude extract purification through systematic static/dynamic adsorption–desorption profiling. Adsorption isotherm analysis demonstrated superior fit with the Langmuir model ($R^2 > 0.94$), while kinetic studies revealed quasi-second-order adsorption mechanics governing D101-SMYAD interactions. The optimized purification protocol was established with the following parameters: sample loading—6 BV at 3 BV/h; impurity removal—3 BV distilled water at 3 BV/h; and target compound elution—3 BV of 90% (*v/v*) ethanol at 3 BV/h. Post-purification yields of target constituents (harpagide, chlorogenic acid, ferulic acid, liquiritin, and harpagoside) ranged from 70.74% to 86.26%, accompanied by a 92% reduction in extract mass per prescription unit (15.072→1.234 g). Chromatic fingerprint similarity evaluation (>0.998) via the 2012 TCM chromatographic system confirmed preservation of the native phytochemical profile. This optimized protocol establishes a reproducible framework for SMYAD preparation standardization, advancing phytopharmaceutical purification methodologies with mechanistic validation.

Author Contributions: Investigation, W.J.; data curation, W.J.; methodology, W.J.; writing—original draft, E.L.; methodology, Y.L.; software, Y.L.; writing—original draft, Y.L.; writing—review and editing, Y.L. writing—review and editing, E.L. and Y.N.; investigation, Y.N.; visualization, Y.N.; conceptualization, Y.G.; resources, Y.G.; writing—review and editing, Y.G.; supervision, Y.G. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data presented in this study are available in the article.

Conflicts of Interest: Author Erhu Liu was employed by the company Shijiazhuang Woguang Technology Co., Ltd. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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