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Designed modification on macroporous resin for extracting sesamin from sesame oil

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ABSTRACT

Macroporous resin was developed to extract sesamin from sesame oil. In order to improve adsorption capacity of macroporous resin, some special compounds were selected to be grafted to macroporous resin. The main factors affecting resin modification were studied and the characteristics of resins were analyzed. High-performance liquid chromatography (HPLC) was used to test the adsorption capacity of resins. Fourier transform infrared spectrometer and Brunauer–Emmett–Teller surface area and pore size analyzer were used to characterize the surface of modified resin. The static and kinetic adsorptions of sesamin on modified resin were studied. Results showed that glycerol was the best grafting compound, and the adsorption ability of macroporous resin increased by 145.16% after modification. The optimum modification condition was the grafting reaction which occurred at 50℃ for 24 h using glycerol as the grafting compound and hydrochloric acid as the catalyst.

Keywords: Sesamin; Macroporous resin; Graft; Resin modification; Sesame oil

1. Introduction

Lignans are one of the major classes of phytoestrogens, which are estrogen-like chemicals and also act as antioxidants. Flax seed and sesame seed are among the known richest sources of lignans [1]. Lignans have been reported to have many physiological functions, for example, lowering blood cholesterol and decreasing liver disease risk [2], and may be associated with improved survival among postmenopausal women with breast cancer [3], and antitumor-promoting activity [4]. Sesame seed is one of the most significant sources of plant lignans. Sesame oil contains mainly sesamin and sesamol [5], and the content is about 0.6–1.2% [6]. Sesame lignans have antioxidative, hypocholesterolemic, and immunomodulatory effects [7–9]. Considering its excellent physiological functions, effective methods for lignan extraction should be developed.

Sesamin has usually been extracted from sesame oil by methanol [10]. The method was created as early as 1949 by Allen L, and there was no improvement in the sesamin extraction method until our report on macroporous resins [11]. There is growing interest in employing macroporous resins to separate bioactive components from crude extracts of herbal raw materials. Macroporous resins are effective because of their unique adsorption properties and advantages including ideal pore structure and various surface functional groups available, low operation costs, less solvent consumption, and easy regeneration. Macroporous resins

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have been successfully applied in the separation of luteolin from pigeonpea leaf extracts, vitexin and isovitexin from pigeonpea extracts, and licorice flavonoids and glycyrrhizic acid from licorice [11,12]. Yin Tian et al. extracted and purified total lignans from rude *Schisandra chinensis* in an aqueous phase with macroporous resins [13]. However, there have been few papers on separating products from the oil phase using macroporous resins.

Adsorption in the oil phase is much more difficult than that in the aqueous phase, because many bioactive components such as lignans tend to dissolve in oil, and oil has higher viscosity which makes adsorbate diffuse difficult. The molecular weight of common edible oil is about 900 Da; so that, it is very difficult for oil to shift in resin.

Modification is a good method to change the adsorption capability of macroporous resins. Usually, resin monomers are first modified and then polymerized to obtain modified resin [14], because monomers are relatively easy to be modified for active groups in monomers. If a specific compound was to be grafted to the resin, modifying the monomer may not be an effective method, because a specific compound would react during polymerization. So, grafting to the resin directly may be more reliable.

According to our previous research results, a phenol–formaldehyde-based resin (XAD-7 HP, Rohm and Haas Company) was developed to extract sesamin from sesame oil [11]. It was more efficient than ethanol extraction, and there would be much less pollution, and very little water and organic solvent would be consumed. To improve this method, a better material propylene glycol was selected to be grafted in XAD-7 HP. In a previous study [11], several kinds of organic solvents were selected to extract sesamin from sesame oil, such as methanol, ethanol, isopropanol, butanol, aether, hexane, cyclohexane, propylene glycol, and glycerol. Results showed that the propylene glycol is the best one, so the propylene glycol was expected to be grafted to the resin framework. Considering that the condensation reaction of hydroxyl may occur during grafting, glycerol is a good candidate. If glycerol was selected as the graft compound, when one hydroxyl group of the glycerol bonding with resin framework, its residue would have similar structure with propylene glycol. There are few references about this kind of grafting; hence the optimum way for XAD-7 HP modification was studied in this paper. Fourier transform infrared spectrometer (FTIR) and Brunauer–Emmett–Teller (BET) surface area and pore size analyzer were used to evaluate the characterizations of modified resins.

2. Experimental

2.1. Materials and reagents

Macroporous resin (XAD-7HP) was obtained from the Rohm and Haas Company (USA). Sesamin standard was purchased from Sigma Chemical Company (St. Louis, USA). Methanol (chromatographic pure), N,N-dimethylformamide (DMF, AR), hydrochloric acid (Analysis Rate, AR) and glycerol (AR), cis-butenedioic acid (AR), divinyl triamine (AR), and tetrahydrofuran (THF, AR) were all obtained from the Country Medicine Reagent Limited Company (Shanghai, China). Sesame oil (hot moulding) was obtained from the Shanghai Pansun Sesame Research Institute (Shanghai, China).

2.2. Adsorption of sesamin

The resin was washed with distilled water and ethanol and then dried. The dried resin (5 g) was mixed with sesame oil (100 g). Adsorption was carried out in a shaker (THC-300, Shanghai Yiheng Technology Co., Ltd., Shanghai, China) for 10 h. The concentration of sesamin in sesame oil was determined by high-performance liquid chromatography (HPLC). Each experiment was carried out in triplicate. Comparing adsorption capacities (Eq. (1)) of macroporous resins, the optimum method for modification resin was established. Adsorption capacity of resin is given by:

$$n = M_0(C_0 - C_1)/m \tag{1}$$

where n is adsorption capacity of resin (mg/g resin), m is the mass of resin (g), M_0 is the mass of initial sesame oil (g), C_0 is the sesamin concentration in initial sesame oil (mg/g oil), and C_1 is the sesamin concentration in the sesame oil after adsorption (mg/g oil).

Shimadzu HPLC (Kyoto, Japan) was equipped with SCL-10Avp system controller, SPD-10Avp UV–VIS detector, LC-10Advp pump, CTO-10Asvp Column oven, and Class-VP software. The detailed parameters to measure concentration of sesamin in sesame oil by HPLC were as follows: VP-ODS column (150 mm × 4.6 mm) was used at 30℃; methanol–water (74:26, v/v, volume ratio) was used as mobile phase at the flow rate of 1.0 ml/min; and the detecting wavelength for sesamin was 286 nm.

2.3. Modification of resin

There are five main factors affecting the function of modified resin, such as the type and dosage of grafting compound, type of catalyst, grafting temperature, and grafting time. Modification reaction was processed as follows: 5 g of XAD-7HP resin mixed with DMF in a thermostat shaker for 8 h (which made resin skeleton flexible and grafting reaction easier to occur). Then a catalyst and grafting compound was added. Stirring continued at grafting temperature for some time. The modified resin was washed successively by sufficient deionized water, 1 mol/L HCl, deionized water, 1 mol/L NaOH, deionized water, and alcohol finally, and then dried in vacuum drier (60℃, 1.3 kPa for 4 h).

2.3.1. *Types of grafting compound*

To improve adsorption capacity to sesamin, a modification method to increase resin polarity should be used. Four types of compounds with different polar groups (hydroxyl, carboxyl, amino, and epoxy group) were grafted to resin, respectively. HCl was used as a catalyst and grafting operations were carried out at 40℃, for 10 h in this reaction. The adsorption capacities of modified resins to sesamin were measured.

2.3.2. *Catalysts*

According to reaction mechanism of esterification and related references [15,16], strong acid, weak acid, strong base, and weak base were all possible catalysts. Four kinds of catalysts (HCl, CH_3COOH, KOH, and anhydro-$AlCl_3$) were used as catalyst, respectively. Glycerol was used as modifying compound and grafting operation was carried out at 40℃ for 10 h.

2.3.3. *Dosage of grafting compound*

According to 2.3.1 and 2.3.2, glycerol was selected as graft compound. The 0.5, 1.0, 1.5, 2.0, 2.5, and 3.0 g of glycerol were designed to react with 5.0 g of XAD-7HP resin, respectively, at 40℃ for 10 h. HCl was used as catalyst. Adsorption capacities of modified resins were measured.

2.3.4. *Grafting temperature*

Limited by the property of resin's base material, modifying temperature should be below 80℃. The 5.0 g of XAD-7HP resin was grafted with 2.0 g of glycerol at 20, 30, 40, 50, 60, 70, and 80℃ for 10 h, respectively. HCl was used as catalyst. Adsorption capacities of modified resins were measured.

2.3.5. *Grafting time*

The 5.0 g of XAD-7HP resin was grafted with 2.0 g of glycerol at 40℃ for 0.5, 1–4, 6, 8, 10, 12, 24, and 48 h, respectively. HCl was used as catalyst. Adsorption capacities of modified resins were measured.

2.3.6. *Dosage of catalyst*

The 0.2, 0.4, 0.6, 0.8, 1.2, 1.5, 2.0, 4.0, and 10.0 g of HCl were used as catalyst for grafting, respectively, (5.0 g XAD-7HP resin, 2.0 g glycerol, 40℃ for 10 h). Adsorption capacities of modified resins were measured.

2.4. *Resin physical properties measurement*

Rupture stress of resin was measured by texture analysis instrument (Stable Micro System, Model TA. XT Plus, UK). Model of transducer was P/36R and test velocity was 0.5 mm/min. Single resin pill was pressed in instrument until crushed and the power was recorded automatically. Each experiment was triplicated.

The structural feature of resins was identified by FTIR. The FTIR spectrophotometer (Bruker, Model tensor-37, Germany) was operated under the conditions of spectral width 4,000-400 cm^{-1}, 32 accumulations, 1 gain, 4 cm^{-1} resolution, and signal processing by triangular apodization with potassium bromide pellet method. The pellet was prepared with a mixture of 300 mg of potassium bromide and 5 mg of resin sample.

The surface area and pore size of resin were detected with BET surface area and pore size analyzer (Quantachrome, model NOVA 4200e, USA) at 80℃, N_2 was used as analysis gas.

2.5. *Static adsorption of resin*

To determine the increase scope of modified resin adsorption capacity, 5.0 g modified resin (dry weight basis) was mixed with 100 g sesame oil by oscillator at three different temperatures (15, 30, and 45℃) for 10 h. The concentration of sesamin in sesame oil was analyzed by RP–HPLC–UV. The increase of adsorption capacity (IPAC) of modified resin is calculated by Eq. (2):

$$\mathrm{IPAC} = \frac{q_{e1} - q_{e0}}{q_{e0}} \times 100\% \tag{2}$$

where q_{e1} is the adsorption rate of modified resin (mg/g) and q_{e0} is the adsorption rate of XAD-7HP resin (mg/g).

2.6. *Dynamic adsorption of resin*

The dynamic adsorption test was performed as follows: 2.5 g of modified resin was filled into a column ($\varphi 25 \times 430$ mm) and sesame oil flowed through the column at a rate of 2 mL/min. The sesamin concentration in effluent sesame oil was determined constantly by HPLC until sesamin concentration unchanged. Dynamic adsorption tests were carried out at 15 and 30℃, respectively.

3. Results and discussion

The performance of resin modification could be identified by the adsorption capacity and physical strength of modified resin.

3.1. *Influence of grafting compound*

The adsorption capacity of modified resins grafted with four types of grafting compound was calculated according to Eq. (2). As shown in Table 1, increased polarity of resin was helpful for improving resin adsorption. In a former research, we found that propylene glycol was better than methanol in extracting sesamin from sesame oil. XAD-7HP resin has carbonyl in structure, if carbonyl could be induced as hydroxyl by catalyst and condensed with hydroxyl in glycerol, propylene glycol will successfully grafted in resin.

The rupture stress values of modified resins were illustrated in Table 2. There was no significant difference on resin rupture stress whether after modifying or modified by different grafting compounds. It demonstrated that all modification reactions we designed did not damage resin's structure. Because both glycerol and propylene glycol are edible, glycerol was the safest grafting compound among four compounds and was efficient too, it was chosen as grafting compound in the following experiments.

Table 1
IPAC (%) of modified resins grafted with four grafting compounds

Grafting compound (with polar groups)	IPAC (%)
Glycerol (hydroxyl)	122.8 ± 3.7
Cis-butenedioic acid (carboxyl)	120.0 ± 3.4
Divinyl triamine (amino)	121.0 ± 3.4
THF (epoxy group)	111.1 ± 3.3

Table 2
Failure stresses of modified resins grafted with four grafting compounds

Grafting compound	Failure stresses (g/pill of resin)
Blank	590.5 ± 2.5
Glycerol	632.5 ± 3.0
Cis-butenedioic acid	625.0 ± 3.0
Divinyl triamine	606.0 ± 2.5
THF	638.0 ± 2.5

3.2. *Optimization of resin modification*

3.2.1. *Influence of catalyst*

The change of adsorption capacity using four types of reagents was calculated according to Eq. (2) and the results are shown in Table 3. It shows obviously that only strong acid (HCl) could improve adsorption capacity of modified resin, while the other three kinds of reagents decreased adsorption capacity. Hence, HCl was used as catalyst in our grafting reaction.

3.2.2. *Influence of glycerol dosage*

As shown in Fig. 1(a), the adsorption capacity of modified resin increased with the glycerol amount from 0.5 to 2.0 g. But it was nearly no more changed when glycerol amount was more than 2.0 g. The 2.0 g of glycerol was considered to be an optimum dosage.

3.2.3. *Influence of grafting temperature*

Temperature was an important factor for the stability of the porous structure, macroporous structures of the resin would collapse under high temperature [17]. Modified temperature was controlled below 80℃ due to the property of resin fundamental material. As shown in Fig. 1(b), the adsorption capacity of modified resin which grafted at temperature above 40℃ was much higher than that below 40℃, whereas there was still a little discrepancy at the range between 40 and 80℃. So the optimum grafting temperature should be confirmed by orthogonal experiments.

Table 3
IPAC (%) of resins grafted with four reagents as catalysts

Reagent	IPAC (%)
HCl	125.4 ± 5.0
CH_3COOH	−10.2 ± 4.5
KOH	75.9 ± 5.3
Anhydro-$AlCl_3$	51.7 ± 4.2

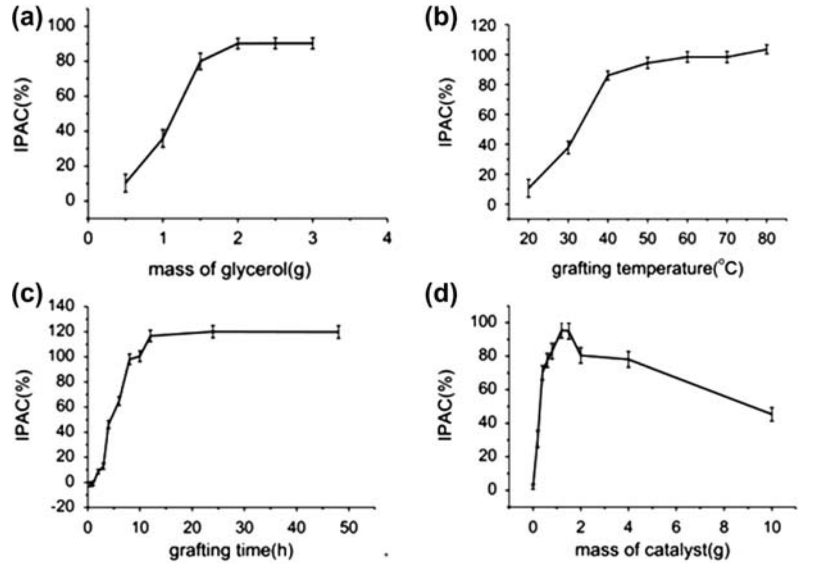

Fig. 1. Influence of grafting factors on IPAC (%): (a) mass of glycerol, (b) grafting temperature, (c) grafting time, and (d) mass of catalyst.

Table 4
Factor level in orthogonal experiments

Levels	Factors				
	A	*B*	*C*	*D*	*E*
	m_{HCl} (g)	*T* (℃)	time (h)	$m_{glycerol}$ (g)	m_{resin} (g)
1	0.6	40	8	2.0	5.0
2	0.8	50	10	2.0	5.0
3	1.2	70	12	2.0	5.0
4	1.5	80	24	2.0	5.0

3.2.4. Influence of grafting time

From Fig. 1(c), it can be concluded that the grafting time was also an important factor. The adsorption capacity of modified resin increased by about 1.2 times from grafting time of 0.5 to 12 h, but increased little from 12 to 48 h.

3.2.5. Influence of catalyst dosage

As indicated in Fig. 1(d), the adsorption capacity increased with the increment of HCl dosage, until the dose came to 1.2 g, while it decreased rapidly after that. The reason may be that low dosage of HCl was not enough for grafting reaction, but excessive amount of HCl could destroy resin structure. The 1.2 g of HCl was considered as a suitable dosage.

3.2.6. Orthogonal experiments of influent factors

In order to optimize the modification reactions, orthogonal design experiments ($L_{16}4^5$) were carried out as follows: glycerol as modifying reagent, concentrated hydrochloric acid (12 N) as catalyst, and reaction temperature and time were another two factors. The results are shown in Tables 4 and 5.

According to the experiments, the optimum schedule was 5.0 g resin grafted with 2.0 g glycerol at 50℃ for 24 h and concentrated hydrochloric acid (0.8 g) was used as catalyst. The adsorption capacity of modified resin in this way was found to be increased by 145.16%. This modified resin was coded as ZZGOH.

3.3. Structural characterization of modified resin

Compared with the FTIR spectra of resin ZZGOH and XAD-7HP (Fig. 2), for ZZGOH FTIR spectra, a broad and bifurcate band was located at 3,440 cm^{-1}. The vibration of hydroxyl strengthened and infrared absorption was enhanced, which meant the proportion increase of hydroxyl in resin. Stronger absorption on methylene (at 1,485 cm^{-1}) and methyl groups (at 1,384 cm^{-1}) occurred, and also C–H band occurred at 2,965 cm^{-1}. All these indicated that the grafting reaction was occurred between resin and glycerol.

The physical characterization of resins is summarized in Table 6. Results demonstrated that the physical properties of resin changed little after modification treatment. It also demonstrated that the changes in chemical structure were the main factor. Although BET specific surface area of ZZGOH is higher than XAD-7HP, it is not the main reason results in its adsorption capacity improving, because it had been found in other experiments that modified XAD-7HP resin had a higher specific surface area (509 m^2/g),

Table 5
Orthogonal experimental results

Factors	*A*	*B*	*C*	*D*	*E*	IPAC (%)
1	1	1	1	1	1	121.2
2	1	2	2	2	2	128.6
3	1	3	3	3	3	123.6
4	1	4	4	4	4	131.4
5	2	1	2	3	4	130.2
6	2	2	1	4	3	133.3
7	2	3	4	1	2	126.8
8	2	4	3	2	1	138.5
9	3	1	3	4	2	122.8
10	3	2	4	3	1	135.0
11	3	3	1	2	4	133.6
12	3	4	2	1	3	124.5
13	4	1	4	2	3	125.9
14	4	2	3	1	4	128.9
15	4	3	2	4	1	131.6
16	4	4	1	3	2	127.1
*k*1	126.20	125.03	128.80	125.35	131.58	
*k*2	132.20	131.45	128.73	131.65	126.33	
*k*3	128.98	128.90	128.45	128.98	126.83	
*k*4	128.38	130.38	129.78	129.78	131.03	
R	6.00	6.43	1.33	6.30	5.25	

B>*A*>*C* Optima schedule: A2B2C4.

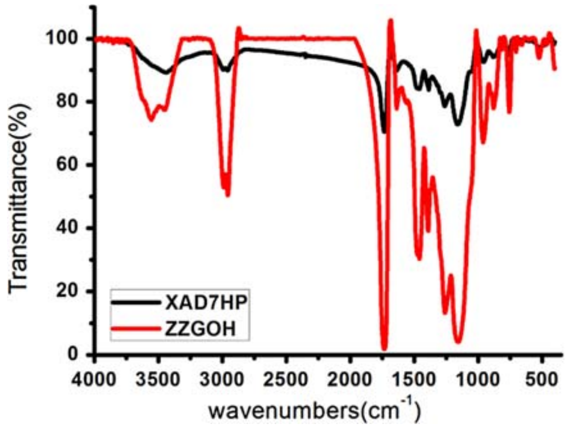

Fig. 2. FTIR spectra of XAD7-HP resin and ZZGOH resin.

Table 6
Physical characteristics of XAD-7HP resin and ZZGOH resin

Characteristics	XAD-7HP	ZZGOH
Polarity	Semipolarity	Semipolarity
BET specific surface area (m^2/g)	450	560
Pore volume (cm^3/g)	1.20	1.12
Particle size (mm)	0.56–0.71	0.56–0.71
Average pore diameter (nm)	8.75	8.55
Color	White	White

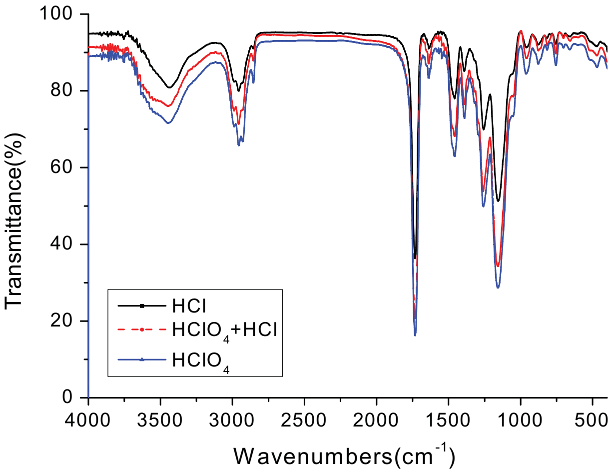

Fig. 3. FTIR spectra of resins with different catalysts.

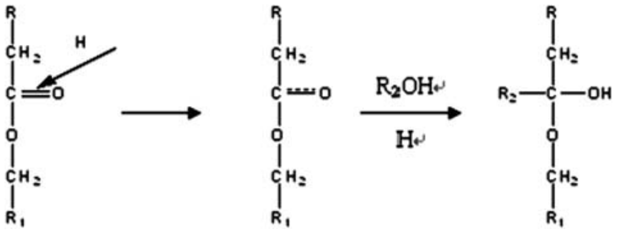

Fig. 4. Possible catalytic mechanism in resin structural modification processing.

but lower adsorption capacity than original XAD-7HP (450 m^2/g).

3.4. Modification mechanism

As shown in Table 3, catalysts played a main role in modifying reaction. In order to verify the roles of H^+ and acid radical, same mole of acid (0.8 g HCl, 2.2 g $HClO_4$, and 0.4 g HCl + 1.1 g $HClO_4$) was used as catalyst, respectively. The FTIR spectra of modified resins were illustrated in Fig. 3. It seemed that the relative adsorption intensities of C–H bond, methylene, and methyl were all strengthened with the acidic enhancement, which may be due to the effect of H^+ on the resin matrix. Because the chlorine could not be analyzed by atomic absorption, it was not clear whether

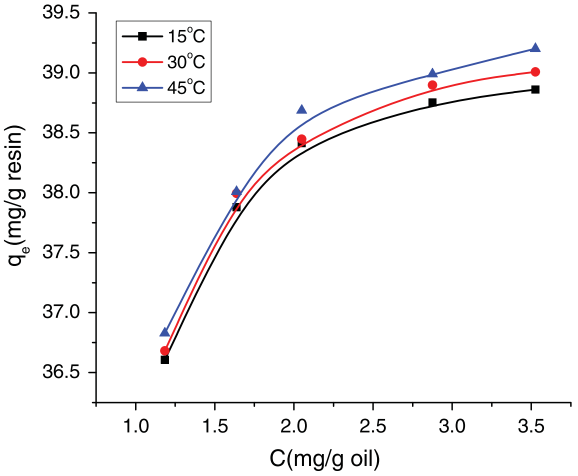

Fig. 5. Three adsorption isotherms of ZZGOH resin static adsorption vs. the concentration of sesame oil.

Table 7
The relevant parameters for the absorption of sesamin on ZZGOH resin

Resin	T (℃)	Langmuir equation		
		K_L	q_m	${R_L}^2$
ZZGOH	15	9.19	40.32	0.964
	30	9.15	40.49	0.973
	45	8.79	40.65	0.975

acid radical participated in catalytic reaction or not. In order to make sure the role of acid radical, strong acid HNO_3 was used as catalyst instead of HCl in same mole. Elemental analysis showed the content of nitrogen in resin was below only 0.3% during entire reaction processing, and it indicated that nitrogen did not cross-link to the resin matrix. It also meant that acid radical did not participate in resin modification. Based on the summarized above results, it is H^+ that played catalyst role in resin structural modification processing. The possible mechanism is illustrated in Fig. 4.

3.5. Identity of modified resin adsorption

3.5.1. Adsorption isotherm of static adsorption

Adsorption isotherms at different temperatures are shown in Fig. 5. Some classical equations can very well describe the different types of adsorption. The adsorption isotherm data obtained in this study were verified by classical equation. Langmuir equation (Eq. (3)) was matched with our adsorption isotherms, with the correlation coefficient R^2 of 0.97 (shown in Table 7).

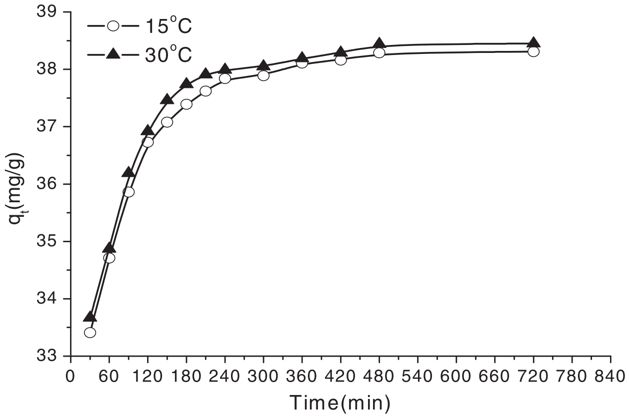

Fig. 6. Influence of temperature on adsorption kinetic.

$$\frac{1}{q_e} = \frac{1}{K_L q_m c_e} + \frac{1}{q_m} \tag{3}$$

where K_L is the adsorption equilibrium constant, q_m is an empirical constant, and q_e is the adsorption quantity of resin at sesamin concentration in c_e.

Usually, adsorption rate was inversely proportional to the temperature for adsorption in the aqueous phase. But, in a much higher viscosity oil phase at a relative low temperature range (lower than 45℃), adsorption quantity of sesamin increased with temperature raise. The possible reason is that sesame oil is very viscous at low temperature. Therefore, the diffusion of sesamin in sesame oil to resin was hampered. A relative higher temperature can reduce viscosity of sesame oil effectively, but it does not increase desorption of sesamin significantly.

3.5.2. Adsorption kinetics

Adsorption kinetics curves are illustrated in Fig. 6. It shows that, the adsorption kinetics of sesamin on ZZGOH resin followed the exponential decay model well. The fitting equations of the both adsorption kinetics curves are as follows:

$$Y = -6.94363e^{-x/84.05895} + 38.2324, R^2 = 0.99705 \tag{4}$$

$$Y = -7.06202e^{-x/77.23226} + 38.3569, R^2 = 0.99436 \tag{5}$$

where Eq. (1) was at 15℃ and Eq. (2) was at 30℃.

While the correlation coefficients were both more than 0.99, the adsorption in column could obtain more than 95% of equilibrium adsorption. All these indicated that ZZGOH resin has good adsorption capacity in the oil phase [18].

4. Conclusions

XAD-7HP resin was successfully grafted with glycerol to increase its polarity, in order to strengthen the adsorptive affinity to polar materials. The efficiency of modified resin ZZGOH was 2.5 times of original resin in adsorbing sesamin from sesame oil. According to the experiments, the optimum schedule was that 5.0 g of resin was grafted with 2.0 g of glycerol at 50℃ for 24 h, concentrated hydrochloric acid (0.8 g) was used as catalyst. The adsorption capacity of modified resin in this way was found to have increased by 145.16%. This research offered us an idea that resin can be modified according to predesign and applied in oil system, which will enlarge application scope of macroporous resin in adsorbing bioactive compounds.

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