Hydroquinone Modified Hyper-*Cross*-Linked Resin to be Used as a Polymeric Adsorbent for Adsorption of Salicylic Acid from Aqueous Solution

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ABSTRACT: Hydroquinone modified hyper-*cross*-linked resin (HJ-Y15) was prepared from macroporous cross-linked chloromethylated polystyrene and the adsorption behaviors of HJ-Y15 for salicylic acid were studied from aqueous solution. The results indicated that the surface of HJ-Y15 was modified by formaldehyde carbonyl, quinone carbonyl, and phenolic hydroxyl groups. Freundlich model was suitable for characterizing the isotherms and the adsorption was shown to be an exothermic, spontaneous, and more ordered process. The pseudosecond-order rate equation was appropriate for describing the kinetic curves and the intraparticle diffusion was the rate-controlling step. Hydrogen bonding between formaldehyde carbonyl groups of HJ-Y15 and carboxyl groups of salicylic acid was one of the primary driving forces for the adsorption. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 121: 3717–3723, 2011

Key words: hyper-*cross*-linked resin; adsorption; hydrogen bonding

INTRODUCTION

Salicylic acid is an important fine chemical and pharmaceutical intermediate, it is often employed to produce medicines like Aspirin, Lopirin, Fenamifuril, Diflunisal, Salicylamide, and Benorylatum.[1] Salicylic acid at a low concentration (0.2–1.5%) is a well-known cosmetic, it can get rid of horniness, deflate pore and wipe off wrinkle. On the other hand, salicylic acid at a high concentration (higher than 2%) is bad for the health, it can result in headache, nausea, and skin sensibility, even it can affect the liver and kidney.[2] Consequently, developing an efficient method for removal and reuse of salicylic acid from aqueous solution is significant.

Adsorption is an effective physical method for removal of aromatic compounds.[3,4] Activated carbon, as a typical adsorbent for removal of aromatic compounds, has a very large adsorption capacity for aromatic compounds due to its high specific surface area and predominant micropores.[5] However, the mechanical strength and effective regeneration are the two serious problems which limit the field application for activated carbon. Synthetic polymeric adsorbent, on the other hand, has attracted many attentions in recent years due to its excellent mechanical strength, diverse chemical structure and convenient regeneration for repeated use and hence has been a potential alternative for activated carbon.[6–9]

Davankov synthesized a kind of hyper-*cross*-linked polystyrene using bi-functional crosslinking reagents and Friedel-Crafts catalysts from linear polystyrene or low crosslinked polystyrene.[10,11] According to this method a great deal of rigid methylene crosslinked bridges are formed between the polymeric chains, and thus the polymeric skeleton of the prepared hyper-*cross*-linked polystyrene is greatly reinforced. The synthesized hyper-*cross*-linked polystyrene has high specific surface area and predominant mesopores, and thus it exhibits excellent adsorption behaviors for nonpolar and weakly polar aromatic compounds.[12,13] To improve the adsorption behaviors of this kind of resin for polar aromatic compounds, three effective techniques such as introduction of polar monomer in copolymers, employment of polar compound as the crosslinking reagent, introduction of polar aromatic compounds in the Friedel-Crafts reaction are frequently applied.[14–17] The results indicate that the retention of the prepared resins for polar aromatic compounds is greatly enhanced.[18,19] If macroporous low cross-linked chloromethylated polystyrene is used as the

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reactant and hydroquinone is added in the Friedel-Crafts reaction, the Friedel-Crafts reaction between hydroquinone and chloromethylated polystyrene will also be involved, and hydroquinone should be uploaded onto the surface of the obtained hyper-*cross*-linked resin and the adsorption behaviors of the obtained hyper-*cross*-linked resin for polar aromatic compounds like salicylic acid should be improved. To the best of our knowledge, no literature focuses on hydroquinone modified hyper-*cross*-linked resin up to now.

In this study, a novel hydroquinone modified hyper-*cross*-linked resin HJ-Y15 is prepared from macroporous low crosslinked chloromethylated polystyrene by adding hydroquinone (15% relative to the mass of chloromethylated polystyrene) in the Friedel-Crafts reaction, the structure of HJ-Y15 is thereafter characterized and the adsorption behaviors of HJ-Y15 for salicylic acid are studied from aqueous solution. The adsorption thermodynamics and kinetics are analyzed and the possible interaction between HJ-Y15 and salicylic acid is investigated by Fourier transform infrared (FTIR) spectroscopy.

EXPERIMENTAL

Materials

Macroporous crosslinked chloromethylated polystyrene was purchased from Langfang Chemical Co. Ltd. (Hubei province, China), its chlorine content was 17.3% and specific surface area was 28 m^2/g. Salicylic acid applied as the adsorbate in this study was an analytical reagent and used without further purification. Anhydrous zinc chloride was employed as the catalysts in the present study. Before using, it was kept cauterant on an electric cooker till it was free from water.

Synthesis of HJ-Y15

As shown in Scheme s1, 40.0 g (accuracy: 0.1 g) of chloromethylated polystyrene was swollen by 120 mL (accuracy: 1 mL) of nitrobenzene, and 6.0 g of hydroquinone (15% relative to the mass of chloromethylated polystyrene) was dissolved by nitrobenzene and added into the reaction mixture. At a moderate agitating speed, 4.0 g of anhydrous zinc chloride was added into the flask at 323 K (accuracy: 0.2 K). After the added zinc chloride was completely dissolved, the reaction mixture was evenly heated to 388 K within 1 h. The hydroquinone modified hyper-*cross*-linked resin HJ-Y15 was synthesized after keeping the reaction mixture for about 8 h at 388 K.

Adsorption isotherms

About 0.1000 g (accuracy: 0.0001 g) of the resin was weighed accurately and 50 mL (accuracy: 0.01 mL) of salicylic acid aqueous solution with known initial concentration were added into a 100 mL of conical flask. The initial concentration of salicylic acid, C_0 (mg/L), was set to be about 100, 200, 300, 400, and 500 mg/L. 0.1 mol/L of hydrochloric acid and 0.1 mol/L of sodium hydroxide were applied to adjust the solution pH. Then the flasks were shaken at a speed of 150 revolutions per minute (rpm) at a desired temperature (298, 308, and 318 K) for about 24 h. After the adsorption reached equilibrium, concentration of the equilibrium salicylic acid solution, C_e (mg/L), was measured and the equilibrium adsorption capacity, q_e (mg/g), was calculated as[8]:

$$q_e = \frac{(C_0 - C_e)V}{W} \tag{1}$$

where V was volume of the solution (L) and W was mass of the resin (g).

Adsorption kinetic curves

For measurement of the time resolved salicylic acid uptake on HJ-Y15, about 1.0000 g (accuracy: 0.0001 g) of resin and 500 mL of salicylic acid solution at a concentration of 1037.5 mg/L were quickly introduced into a conical flask and continuously shaken at a speed of 150 rpm. Totally, 0.5 mL of solution was sampled at different time intervals and concentration of the residual salicylic acid was determined until adsorption equilibrium was reached, the adsorption capacity at contact time t, q_t (mg/g), was calculated as[20]:

$$q_t = \frac{(C_0 - C_t)V}{W} \tag{2}$$

here C_t was the concentration of salicylic acid at contact time t (mg/g).

Analysis

The pore structure (specific surface area, pore volume, porosity, pore size distribution, etc.) of the resin was determined via N_2 adsorption-desorption curves at 77 K by a Micromeritics Tristar 3000 surface area and porosity analyzer. FTIR spectroscopy of the resin in the range of 500–4000 cm^{-1} was collected by KBr disks on a Nicolet 510P FTIR instrument. The concentration of salicylic acid was measured by UV spectrometry at a wavelength of 296.5 nm.

RESULTS AND DISCUSSION

Structure of HJ-Y15

The specific surface area and pore volume of HJ-Y15 are measured to be 446.3 m^2/g and 0.3102 cm^3/g, respectively. Figure s1(a) describes the N_2 adsorption-desorption isotherms of HJ-Y15. The adsorption isotherm seems close to Type-II isotherm. At the initial part of the isotherm at a relative pressure below 0.10, the adsorption capacity increases rapidly with increment of the relative pressure, proving that micropores are existent (*t*-plot micropore area: 248.4 m^2/g, *t*-plot micropore volume: 0.1355 cm^3/g). The visible hysteresis loop of the desorption isotherm indicates that mesopores are also existent. These analyses agree with the pore diameter distribution in Figure s1(b).

The IR spectra of HJ-Y15 and the chloromethylated polystyrene are displayed in Figure s1(c). A strong C-Cl stretching vibration of CH_2Cl groups at 1265 cm^{-1} for the chloromethylated polystyrene is greatly weakened after the Friedel-Crafts reaction,[21] while there appears a moderate vibration with frequency at 1699 cm^{-1} for HJ-Y15, this vibration can be assigned to the C=O stretching of formaldehyde carbonyl groups and which may be from the oxidation of benzyl chloride of the chloromethylated polystyrene.[22] Another moderate band appears at 1653 cm^{-1}, and it is related to C=O stretching of quinone carbonyl groups.[23] This implies the uploaded hydroquinone is partly oxidized. In addition, a medium-intensity absorption band appears at 3532 cm^{-1} and it can be assigned to the O—H stretching of hydroquinone. The results of the IR spectrum reveal that formaldehyde carbonyl, quinone carbonyl and phenolic hydroxyl groups are uploaded on the surface of HJ-Y15 successfully.

Effect of the solution pH on the adsorption

Figure 1 displays the effect of the solution pH on adsorption of salicylic acid onto HJ-Y15 from aqueous solution. It is observed that the adsorption capacity decreases gradually with increment of the solution pH. Salicylic acid is a weak acid and the initial pH of salicylic acid solution is measured to be 3.11 in this study. Salicylic acid can be ionized in aqueous solution (Scheme s2), as the solution pH is higher than 3.11 (adding sodium hydroxide in the solution), salicylic acid will be ionized as negative ions (a) and (b). While as the solution pH is lower than 3.11 (adding hydrochloric acid in the solution), the ionization will be restrained, and more salicylic acid molecules will be gained. The fact that the adsorption capacity of salicylic acid onto HJ-Y15 decreases with increment of the solution pH implies that the molecular form of salicylic acid is favorable for the adsorption.[21] In addition, it seems that the adsorption capacity is comparatively close and a flat roof appears as the solution pH is in the range of 4.9–9.4, which may be from the reason that the negative ion (a) can also be adsorbed on HJ-Y15.

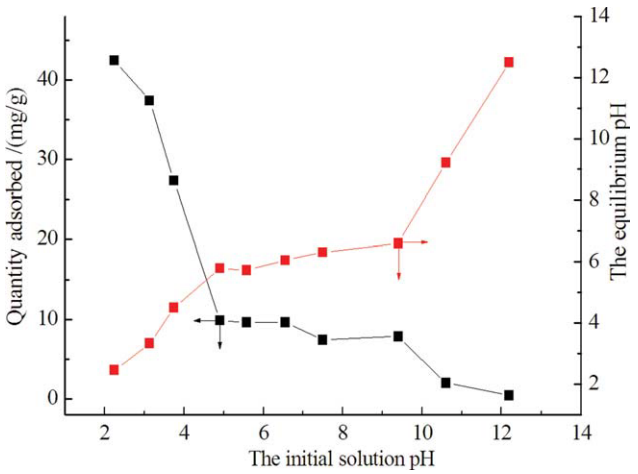

Figure 1 Effect of the solution pH on the adsorption of salicylic acid on HJ-Y15 from aqueous solution. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Compared the equilibrium solution pH with the initial solution pH, it can be observed that the equilibrium solution pH firstly increases and then decreases relative to the initial solution pH. Moreover, the equilibrium solution pH keeps steady and a flat roof also appears as the initial solution pH is in the range of 4.9–9.4, which may be relative to the ionization and hydrolyzation equilibrium of the negative ions (a) and (b) of salicylic acid, and the specific reason is explained underway.

Effect of concentration of phenol in the solution on the adsorption

Phenol is the basic material to prepare salicylic acid and it is inevitably existent in the wastewater of salicylic acid. Figure s2 shows the effect of phenol on the adsorption and it is obvious that the adsorption capacity of salicylic acid onto HJ-Y15 firstly increases and then decreases with increasing of the concentration of phenol. Phenol is a competitive adsorbate for salicylic acid, and which will weaken the adsorption of salicylic acid on HJ-Y15 at a low concentration.[24] Conversely, solubility of phenol in water is much higher than salicylic acid (solubility of phenol and salicylic acid in water at room temperature are 9.3 and 0.22g/100 mL H_2O).[21] As phenol is added in the solution of salicylic acid, more water molecules are "trapped" to surround phenol, which increases the hydrophobicity of salicylic

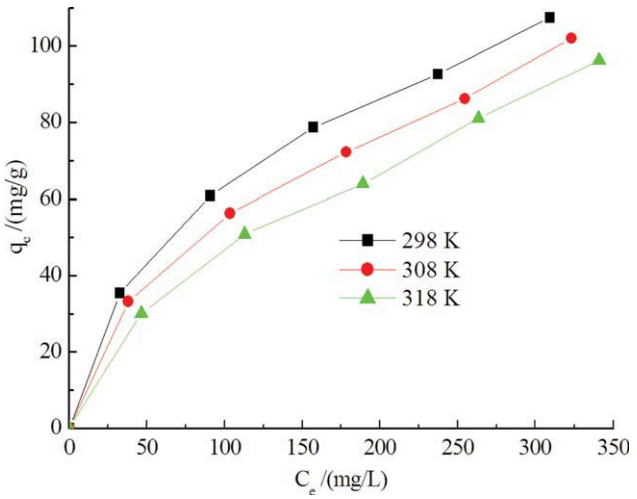

Figure 2 Adsorption isotherms of salicyclic acid onto HJ-Y15 from aqueous solution with temperature at 298, 308 and 318 K, respectively. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

acid.[25] Hydrophobic interaction is shown to be the main driving force for the adsorption of aromatic compounds onto hyper-*cross*-linked resin from aqueous solution.[26] The more hydrophobic the adsorbate is, the more enhanced the adsorption of the adsorbate onto the hyper-*cross*-linked resin from aqueous solution is.[27] Therefore, the increased hydrophobicity of salicylic acid from aqueous solution will induce an enhanced adsorption onto HJ-Y15.

Adsorption isotherms

Figure 2 displays the adsorption isotherms of salicylic acid onto HJ-Y15 with the temperature at 298, 308, and 318 K. All of the isotherms are Type-I ones and the adsorption capacity decreases with increasing of the temperature, suggesting that the adsorption is an exothermic process.[25] Langmuir and Freundlich models are the two typical models to describe the adsorption process. Langmuir model can be given as[8,17]:

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

here q_m is the maximum adsorption capacity (mg/g), and K_L is a constant (L/g).

Freundlich model can be rearranged as[20]:

$$\log q_e = \frac{1}{n} \log C_e + \log K_F \tag{4}$$

where K_F [(mg/g)(L/mg)$^{1/n}$] and n (dimensionless) are the constants. Table s1 summarizes the regression equations, parameters K_L, K_F, n and the correlation coefficients R^2 by Langmuir and Freundlich models. Freundlich model can fit the isotherms since $R^2 > 0.99$, suggesting the adsorption is a multilayer adsorption process and HJ-Y15 possesses heterogeneous adsorption sites.[20]

Adsorption thermodynamics

According to the Clausius-Clapeyron equation[20]:

$$\ln C_e = -\frac{\Delta H}{RT} + C' \tag{5}$$

where C' is the integral constant. By plotting of ln C_e versus $1/T$ (Fig. s3), it is found that the isosters of ln $C_e - 1/T$ can be fitted to straight lines, and ΔH can be calculated from the slopes of the straight lines.

Gibbs free energy can be calculated as[27,28]:

$$\Delta G = -RT \int_0^x q \frac{dx}{x} \tag{6}$$

where ΔG is the Gibbs free energy (kJ/mol), q is the adsorption capacity on the adsorbent (mg/g), and x is the mole fraction of the adsorbed solute in the solution. As the adsorption can be characterized by Freundlich model, incorporating the Freundlich model into eq. (6) will yield:

$$\Delta G = -nRT \tag{7}$$

here n is the characteristic constant in Freundlich model.

Adsorption entropy can be calculated by Gibbs-Helmholtz equation as:

$$\Delta S = \frac{\Delta H - \Delta G}{T} \tag{8}$$

The ΔH, ΔG, and ΔS of salicylic acid adsorbed onto HJ-Y15 are summarized in Table s2. The ΔH is negative, indicating an exothermic process.[25] ΔH decreases with increasing of salicylic acid uptake on HJ-Y15, which is resulted from the surface energetic heterogeneity of HJ-Y15.[29] The ΔG is also negative, indicating that the adsorption is a spontaneous process. The ΔS is negative, revealing that a more ordered arrangement of salicylic acid molecules is shaped on the surface of HJ-Y15.

Adsorption kinetics

Figure 3 is the plots of salicylic acid uptake onto HJ-Y15 versus the contact time at 288, 298, and 318 K. All of the adsorptions can approach equilibrium within 700 min. As the temperature rises from 288 K to 318 K, shorter required time from the beginning to the equilibrium is needed (from 700 min to 300

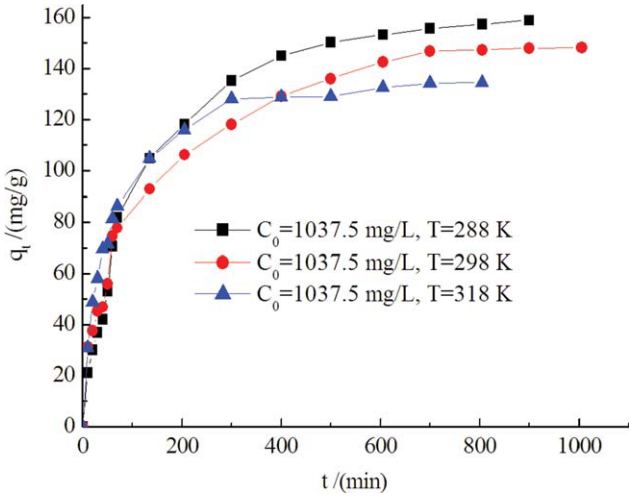

Figure 3 Adsorption kinetic curves of salicyclic acid adsorbed onto HJ-Y15 from aqueous solution with temperature at 288, 298 and 318 K, respectively. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

min) but lower salicylic acid uptake is adsorbed onto HJ-Y15.

Lagergren equation is only suitable for the adsorption kinetics in the beginning process but not the whole one,[30] while pseudosecond-order rate equation by Ho is suitable for the whole adsorption process,[31,32] its linear form is:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{9}$$

here k_2 is the pseudosecond-order rate constant (g/(mg min)). The fitted results in Table s3 indicate that the pseudosecond-order rate equation characterizes the adsorption well due to $R^2 > 0.99$. In particular, the k_2 at 288, 298, and 318 K follows an order as $k_{2\ 318K} > k_{2\ 298K} > k_{2\ 288K}$, accordant with the above observation that a higher temperature induces shorter required time. The initial adsorption rate (h, [mg/(g min)] and half-adsorption time ($t_{1/2}$) with unit of min can be calculated as[33]:

$$h = k_2 q_e^2 \tag{10}$$

$$t_{1/2} = \frac{1}{k_2 q_e} \tag{11}$$

It is observed from Table s3 that h is greater while the $t_{1/2}$ is much shorter at a higher temperature. The apparent activation energy E_a (kJ/mol) can be calculated according to the Arrhenius equation as[33]:

$$\ln k_2 = -\frac{E_a}{RT} + \ln k_0 \tag{12}$$

where k_0 is a constant. By plotting of ln k_2 versus $1/T$, a straight line will be obtained, and E_a can be calculated to be 23.32 kJ/mol.

Adsorption mechanism

It is known that external diffusion (mass transfer from the liquid solution to the adsorbent surface), internal diffusion (or intraparticle diffusion, mass transfer in the pore of the adsorbent) and actual adsorption on the active sites of the adsorbent are the three necessary stages for the adsorption of aromatic compounds onto a porous adsorbent.[34,35] The adsorption mechanism can be determined by plotting of q_t/q_∞ against $t^{1/2}$ according to Fick's second law as[36]:

$$\frac{q_t}{q_\infty} = \frac{6}{r}\sqrt{\frac{Dt}{\pi}} \tag{13}$$

here r is the radius of the polymeric adsorbent (cm) and D is the diffusion coefficient (cm^2/s).

Figure s4 shows the plots of q_t/q_∞ against $t^{1/2}$ and it is evident that they can be classified into at least three stages. The first stage signifies the instantaneous adsorption process, and in this stage the adsorbent is surrounded by a boundary layer of fluid film through which the adsorbate must diffuse prior to external sorption on the adsorbent surface. The second stage reflects the intraparticle diffusion and the third portion is the final equilibrium stage. The intraparticle diffusion coefficient D can be calculated from the slopes of the second portion to be 3.653×10^{-7}, 3.716×10^{-7} and 7.717×10^{-7} cm^2/s with the temperature at 288, 298, and 318 K. The experimental data obtained from this study are examined by the diffusion equation as[37]:

$$t_{1/2} = 0.03 \times r^2/D \tag{14}$$

and the experimental data of D is calculated to be 4.860×10^{-7}, 5.855×10^{-7}, and 1.126×10^{-6} cm^2/s, very close to the calculated ones. According to these results, the rate-controlling step is the intraparticle diffusion.

Possible interaction between HJ-Y15 and salicylic acid

Formaldehyde carbonyl, quinone carbonyl and phenolic hydroxyl groups are uploaded on the surface of HJ-Y15, salicylic acid has phenolic hydroxyl and carboxyl groups, and there should be some specific interaction between the uploaded functional groups of HJ-Y15 and phenolic hydroxyl as well as carboxyl groups of salicylic acid. In this

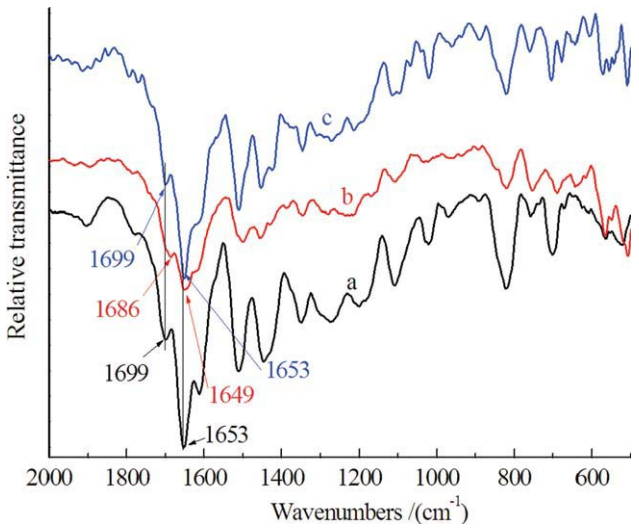

Figure 4 FTIR spectroscopy of HJ-Y15: (a) before adsorption of salicylic acid; (b) after adsorption of salicylic acid; (c) after the adsorbed salicylic acid on HJ-Y15 was desorbed. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

study, FTIR spectroscopy is utilized to examine the shifts of formaldehyde carbonyl and quinone carbonyl groups of HJ-Y15 so that the possible interaction can be clarified. The two main vibrations of HJ-Y15 can be assigned as (Fig. 4): 1699 cm^{-1} (formaldehyde carbonyl) and 1653 cm^{-1} (quinone carbonyl). After adsorption of salicylic acid, the vibrational frequency of formaldehyde carbonyl groups is red-shifted by 13 cm^{-1} while few red-shifts are observed for quinone carbonyl groups (4 cm^{-1}). Moreover, after the adsorbed salicylic acid on HJ-Y15 is desorbed, the vibrational frequency of the formaldehyde carbonyl groups comes back to 1699 cm^{-1}. However, no vibrational frequency shift is observed for some other vibrations like C=C stretching of benzene ring. In common, formation of hydrogen bonding will make some characteristic bands be red-shifted.[38,39] It is deduced that hydrogen bonding between formaldehyde carbonyl groups of HJ-Y15 and carboxyl groups of salicylic acid is involved in the adsorption.

CONCLUSIONS

A novel hyper-*cross*-linked resin HJ-Y15 is prepared successfully, its surface is chemically modified by formaldehyde carbonyl, quinone carbonyl and phenolic hydroxyl groups. Fredulich isotherm model depicts the adsorption isotherms well and the adsorption enthalpy, Gibbs free energy and adsorption entropy are all negative. The pseudosecond-order rate equation characterizes the kinetic curves well and the intraparticle diffusion is the main adsorption mechanism. Formaldehyde carbonyl of HJ-Y15 is helpful for the adsorption and hydrogen bonding is one of the main interactions between HJ-Y15 and salicylic acid.

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