Desalination and Water Treatment
www.deswater.com

doi: 10.1080/19443994.2014.919239

55 (2015) 514–521
June

Taylor & Francis
Taylor & Francis Group

Removal of tartaric acid by gel and macroporous ion-exchange resins

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Received 13 September 2013; Accepted 24 April 2014

ABSTRACT

The objective of this study is to investigate the removal efficiency of tartaric acid from aqueous solution and wine waste by gel and macroporous ion-exchange resins. The resin amount and pH have an important effect on the removal rate. The maximum removal of tartaric acid was achieved at $\text{pH} \geq 4$. Tartaric acid adsorption isotherms were analyzed using Langmuir and Freundlich models. The results obtained have shown that Langmuir model fits well for both resins. Sorption kinetics for these resins fit to Ho pseudo-second-order kinetics model. The hybrid precipitation-cation exchange resin system was used for recovery of tartaric acid from liquid wine wastes. The obtained results showed that hybrid system can be used as an alternative method for the recovery of tartaric acid. The 90% of tartaric acid was recovered by hybrid system.

Keywords: Ion exchange; Purolite A 500; Purolite PFA 400; Tartaric acid; Wine wastes

1. Introduction

Tartaric acid is a white crystalline diprotic organic acid. It is unique in that it is not found in most fruit but is the primary acid component in grapes. It is one of the strongest acids in wine and controls its acidity. Salts of tartaric acid are known as tartrates. They are dihydroxy derivatives of dicarboxylic acid. Naturally occurring tartaric acid is chiral; it means that it has molecules that are not superimposable on their mirror images [1]. Fig. 1 shows the flow diagram of wine making [2].

Tartrates are found in the developing grape as tartaric acid but as the berry ripens the acid content decreases and is replaced by tartrate salts of potassium and calcium. These salts precipitate out during winemaking at various stages.

Wine production industries generate a large amount of waste and by-products. Efficient, inexpensive, and environmentally rational utilization of these wastes and by-products are important for higher profitability and minimal environmental impact. The valuable constituents recovered from wastes used in pharmaceutical, cosmetics, and food industries [3]. Wastewaters from wineries contain tartaric acid. The wastewaters and the skins of grapes from the winemaking process are the only industrial source for tartaric acid production [4]. Tartaric acid and its salts cream of tartar, potassium hydrogen tartrate (KHT) and Rochelle salt (potassium sodium tartrate) are beneficial chemicals, and used in many application

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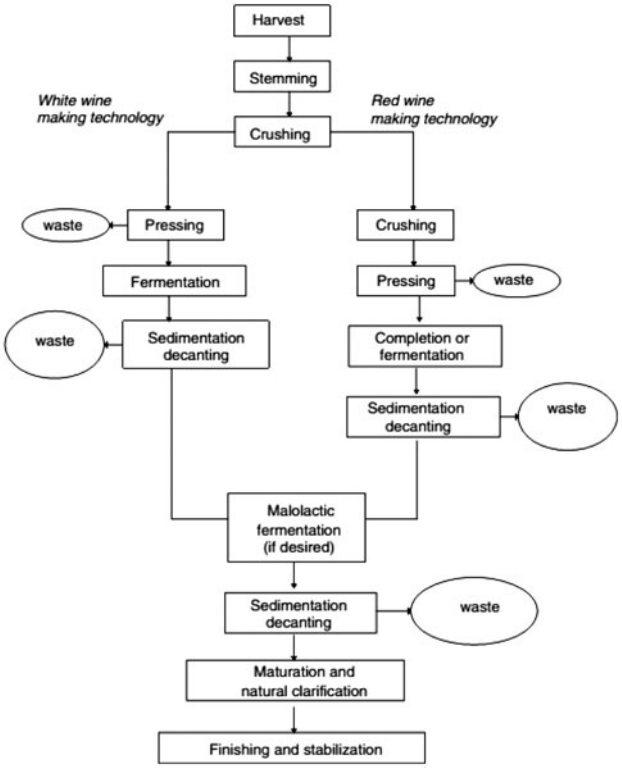

Fig. 1. Wine making flow diagram.
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areas. Tartaric acid is used in food industry and as an emulsifier in baking industry. It is used in the manufacture of fine drug salts, as in effervescent salts, and in cough syrups. Also, it has been utilized in photography, textile and plastics industry, in metals as a chelating agent, in ceramics, and used in electronics industry. Tartaric acid is used for making silver mirrors, in the manufacturing of soft drinks, to provide tartness to foods, in tanning leather, and in making blueprints. It readily dissolves in water and is used in making blueprints [5]. The recovery of tartaric acid as a valuable product from winery wastes and wastewaters is of great interest. Adsorption [4–6], extraction, [7–9], electrodialysis [1,10–12], and ion exchange [3,13–15] are used to recover tartaric acid from wastes. Duca et al. [15], compared the strong base and weak base anion-exchange resins for sorption of tartaric acid.

The aim of our study is to compare the performance of gel and macropore ion-exchange resins for the removal of tartaric acid from aqueous solution. The kinetics performance of resins was also compared. On the other hand, modeling for sorption of tartaric acid was carried out. The second aim is to recover tartaric acid from wine wastes by using precipitation-ion-exchange hybrid process. In this process Calcium tartrate (CaT) was precipitated from waste and after filtration of precipitate; cation exchange resins were used instead of HCl for the dissolving of CaT.

2. Experimental

2.1. Materials

Strong base anion exchange resins; Purolite A 500 (Macroporous) and Purolite PFA 400 (Gel type) were supplied by Purolite Int. Italy. Their characteristics are summarized in Table 1. Model tartaric acid solution was prepared with certain amounts of L(+)-tartaric acid (Merck, Germany). pHs of the solutions were adjusted with NaOH (Merck, Germany) or HCl (Merck, Germany).

2.2. Batch mode sorption studies

2.2.1. Optimum resin dose

Optimum resin dosage was determined by using various amounts of the resin (0.025; 0.05; 0.075 0.10; 0.20; 0.30; 0.40, and 0.50) by contacting with 25 mL of tartaric acid solution (1,000 mg tartaric acid/L, pH 8) equilibrated for 24 h at 25˚C with continuous shaking. After 24 h, the aqueous phase was separated from resins by decantation, and the concentrations of the tartaric acid in the aqueous phase were determined using a UV–vis. spectrophotometer (PG Instrument, PG 80+ model).

The adsorption capacity (q_e, mg/g) and the removal percentage (A, %) were calculated from the following Eqs. (1) and (2) [16]:

$$q_e = V\frac{C_0 - C_e}{m_s} \tag{1}$$

$$A = \frac{C_0 - C_e}{C_0} \times 100 \tag{2}$$

where C_0 and C_e (mg/L) are the initial and final concentrations of tartaric acid in the solution, V is the

Table 1
Physico-chemical properties of resins

Properties	Purolite A 500	Purolite PFA 400
Total capacity (min.) eq/l	1.15	1.3
Temp limit, OH^- form (˚C)	65	60
Moisture retention (%)	53–58	48–54
Specific gravity	1.08	1.08

volume of the solution (L), and m_s is the mass of dry resin (g).

2.2.2. Optimum pH

In order to find the optimum pH; 0.3 g (the optimum amount) of the resins was added to 25 mL 1,000 mg/L of tartaric acid solution at various pH in the 1–11 range equilibrated for 24 h at 25˚C with continuous shaking. After 24 h, the aqueous phase was separated from resins by decantation, and tartaric acid concentration in the solution was analyzed.

2.2.3. Equilibrium adsorption isotherm

For equilibrium adsorption isotherm, 0.3 g of resins was contacted with 25 mL tartaric acid solution with concentrations of 1,000; 2,000; 4,000; 6,000; 8,000; 10,000 mg/L (pH 8) at 25˚C for 24 h under continuous shaking. After 24 h, the aqueous phase was separated from resins by decantation, and tartaric acid concentration in the solution was analyzed (after adequate dilution).

2.2.4. Kinetic experiments

Kinetic tests were performed by contacting resin (12 g) with 1.0 L of tartaric acid solution (1,000 mg/L at pH 8). Solution was stirred with overhead mixer. Tartaric acid concentrations were monitored by taking the samples at prescribed times (1, 3, 5, 10, 15, 30, 45, 60, 90, 120, 180, and 240 min).

2.3. Analysis of tartaric acid

Concentration determination of tartaric acid was carried out by spectrophotometric method as follows: adequate volume of sample solution is transferred to 25 ml volumetric flask and 2 mL 5% (w/v) solution of sodium metavanadat (Alfa-Aesar), and 0.5 mL of concentrated glacial acetic acid (d:1.266 g/ml and 99% w/w) added then flask content was filled with distilled water till the level. The solution was left for 15–20 min at dark for the formation of the stable color. Sample is measured at 490 nm wavelength.

3. Results and discussion

3.1. Optimum wavelength for tartaric acid determination

Determination of tartaric acid by metavanadate method is easy and fast. However, authors of previous studies have used different wavelength for measurement. For instance Mattric and Rice measured at 520 nm [17], Matchett et al. at 515 nm [13], Duca et al. [15] and Fernandes et al. [18] used 490 nm; Hill and Caputi 480 nm [19] for measurement. In our case firstly we found the maximum wavelength for determination. For this 200 mg/L tartaric acid solution was used. The wavelength scanned after blank correction. As can be seen from Fig. 2, the wavelength of maximum absorbance is 490 nm ideal for tartaric acid concentration measurement.

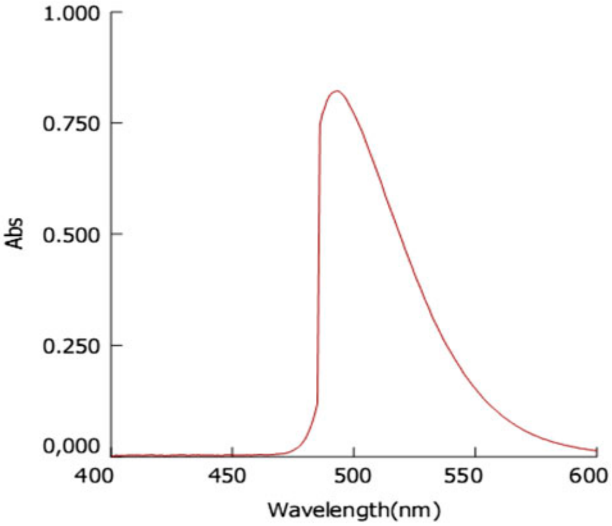

Fig. 2. Absorption spectra of tartaric acid complex with metavanadate.

3.2. Effect of resin dose on tartaric acid removal

To find an appropriate resin amount for complete removal of tartaric acid from aqueous solution, a batch mode sorption study was performed by varying the amount of resins.

Fig. 3 shows the removal of tartaric acid as a function of the resin dosage. The result shows that both the resins have similar performance in the removal of tartaric acid. This may be due to the high ion-exchange capacity of the resins. The experimental results revealed that tartrate removal efficiency increases up to the optimum dosage beyond which the removal efficiency has no change with the resin dosage. As expected, the equilibrium concentration decreases with increasing adsorbent doses for a given initial tartrate concentration, because increasing adsorbent doses provide greater ion-exchange sites for a fixed initial solute concentration [20]. As can be seen from Fig. 3; the optimum resin amount is 0.3 g for both resins.

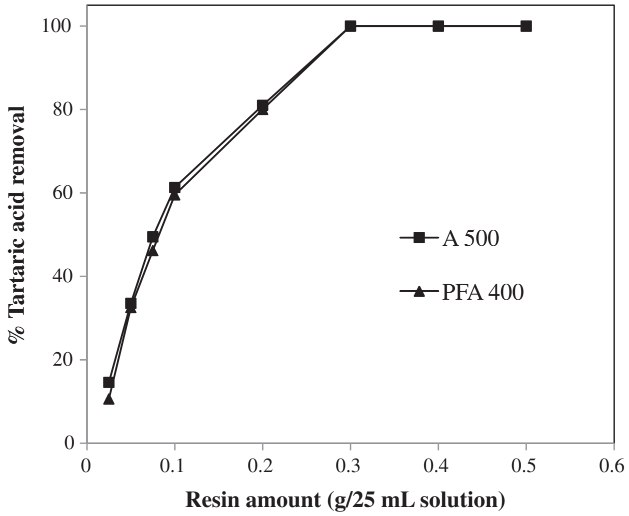

Fig. 3. Effect of resin amount on tartaric acid removal from aqueous solution.

3.3. Effect of pH

To identify the dependency of the ion exchange process; the model solution pH values were varied from 1 to 11 by using HCl or NaOH. The 25 mL of tartaric acid solution (1,000 mg/L) was contacted with 0.3 g resins. Fig. 4 shows the effect of pH on the removal of tartaric acid.

As can be seen from Fig. 4, when the pH of solution is greater than 4, more than 95% removal of tartaric acid was obtained. When pH of solution increases the percent removal of tartaric acid increases. This can be explained as follow; tartaric acid is a weak di-acid. Its pK_a values are 3.0 (pK_a1) and 4.9 (pK_a2) [21]. At low pH values tartaric acid turns its molecular form thus ion-exchange resins cannot remove the tartaric acid. When the pH was increased, tartaric acid starts to dissociate, ionic species forms, and resin can remove hydrogen tartrate or tartrate ions.

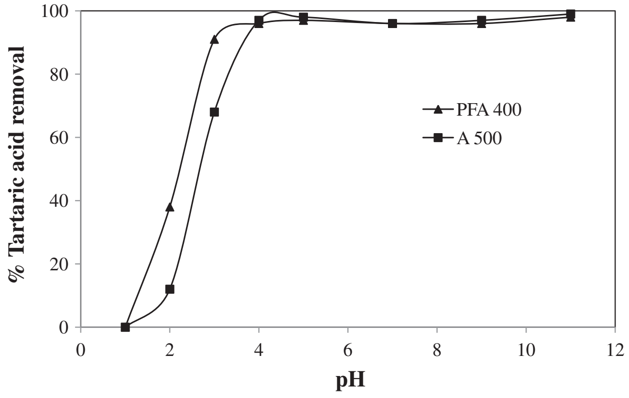

Fig. 4. Effect of pH on the removal of tartaric acid.

The obtained results were compared with literature. Duca et al. [15] used gel type strongly basic and weak basic anion exchange resins. The obtained results showed that tartaric acid can be removed from solution between pH 2.5–5 with strong base resin and pH 2–2.5 with weak base resin. The capacities of weak base and strong base resins are close to resin used in this work (PFA 400 and A 500).

3.4. Adsorption isotherm

3.4.1. Langmuir model

The Langmuir isotherm is a commonly applied model for adsorption on a completely homogenous surface with negligible interaction between adsorbed molecules. The model assumes uniform adsorption energies onto the surface and maximum adsorption depends on saturation level of monolayer [22]. Langmuir model can be represented with the following linear Eq. (3):

$$\frac{C_e}{Q_e} = \frac{1}{bQ_0} + \frac{C_e}{Q_0} \tag{3}$$

where Q_e (mg/g) is the amount of tartaric acid adsorbed per gram of dry resin at equilibrium, C_e is the equilibrium concentration of tartaric acid in the solution (mg/L), Q_0 (mg/g), and b (L/mg) are the Langmuir constants related to the capacity and energy of sorption, respectively.

3.4.2. Freundlich model

The Freundlich model is known as earliest empirical equation and is shown to be consistent with exponential distribution of active centers, characteristic of heterogeneous surfaces [23]. Freundlich equation is (4):

$$\log Q_e = \log K_f + \frac{1}{n}\log C_e \tag{4}$$

where K_f and n are the Freundlich constant that indicate relative capacity and adsorption intensity, respectively.

The experimental data of the ion-exchange equilibrium for the sorption of tartaric acid on the resins tested are depicted in Figs. 5 and 6. The correlation coefficient for the linear regression fit of Langmuir isotherm equation was found to be 0.99 (Fig. 6) for PFA

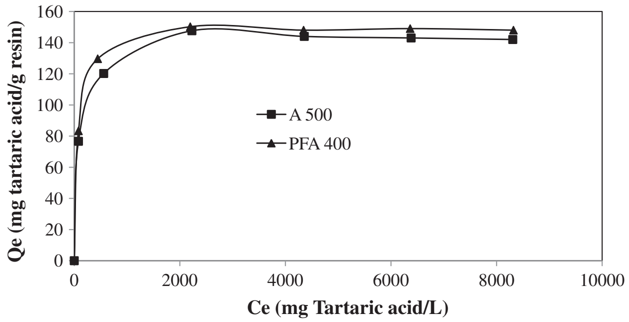

Fig. 5. Relationship between equilibrium concentration and adsorption capacity on uptake of tartaric acid from aqueous solution.

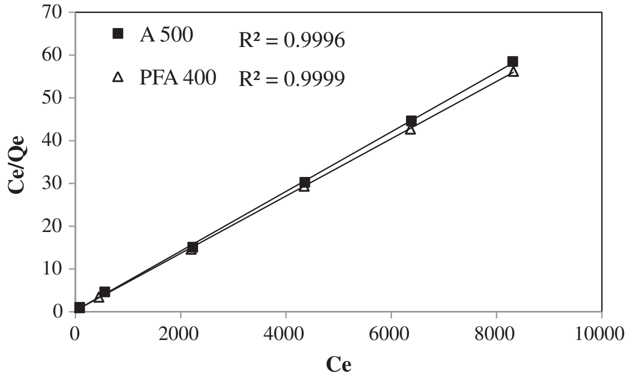

Fig. 6. Linearized form of Langmuir isotherm for Purolite A 500 and PFA 400 resin.

400 and A 500 resins. According to the results, the sorption of tartaric acid on ion-exchange resins is more of monolayer sorption rather than sorption on a surface having heterogeneous energy distribution.

As can be seen from Table 2, the PFA 400 and A 500 resins have nearly the same maximum sorption capacity of 149 and 143 mg tartaric acid/g dry resin.

The shape of the Langmuir isotherm can be used to predict whether a sorption system being favorable or unfavorable in a batch adsorption process. Accordingly, the essential features of the Langmuir isotherm was expressed in terms of a dimensionless constant called the equilibrium parameter, E_p, which is defined by the following relationship (Eq. (5)) [24]:

$$E_p = \frac{1}{1 + bC_0} \tag{5}$$

where E_p = is a dimensionless equilibrium parameter or separation factor, b = constant from Langmuir equation, C_0 = initial tartaric acid concentration

The parameter, E_p, indicates the shape of the isotherm and nature of the sorption process as given below [24]:

Values of E_p	Type of isotherm
$E_p > 1$	Unfavorable isotherm
$E_p = 1$	Linear isotherm
$E_p = 0$	Irreversible Isotherm
$0 < E_p < 1$	Favorable isotherm

In all worked concentrations, E_p values was higher than 0 and lower than 1. The results obtained show that sorption of tartaric acid by resins are favorable.

3.5. Kinetic of resins

In order to predict the mechanism involved in the sorption process, Lagergren pseudo-first and pseudo-second-order models was used. The sorption kinetics following the pseudo-first-order model is given by (6) [25].

$$\frac{dq_t}{d_t} = k_1(q_e - q_t) \tag{6}$$

where q_t and q_e represent the amount of adsorbed species (mg/g) at any time t and at equilibrium time, respectively, and k_1 represents the sorption rate constant (min^{-1}). Integrating Eq. (6) with respect to boundary conditions t = 0 to t = t and q_t = 0 to q_t = q_t, one obtain (7)

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1 t}{2.303} \tag{7}$$

Table 2
Isotherm constants for tartaric acid sorption by ion-exchange resins

	Langmuir isotherm constants			Freundlich isotherm constants		
Resin	Q_0 (mg/g)	b (L/mg)	R^2	K_f (mg/g)	n	R^2
PFA 400	149.25	0.0243	0.99	47.48	0.145	0.88
A 500	142.86	0.0267	0.99	38.89	0.166	0.93

Sorption rate constant k_1 (min^{-1}) can be calculated from the plot of log (q_e–q_t) vs. time.

If the rate of sorption is a second-order mechanism, the pseudo-second-order kinetic rate equation is expressed as (8) [25]:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \tag{8}$$

where k_2 is the pseudo-second-order rate constant (g/mg min), q_e and q_t are the amount of adsorbed species (mg/g) at equilibrium and at time t. By changing the variables in Eq. (8) one get Eq. (9).

$$\frac{dq}{(q_e - q_t)^2} = k_2 dt \tag{9}$$

boundary conditions t = 0 to t = t and q_t = 0 to q_t = q_t, one obtains the final form (10).

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{10}$$

If pseudo-second-order kinetics is applicable, the plot of t/q_t against t of Eq. (10) should give a linear relationship, from which q_e and k_2 (g/mg min) can be determined from the slope and intercept of the plot and there is no need to know any parameter beforehand. Constant k_2 is used to calculate the initial sorption rate r; at $t \rightarrow 0$, as follows (11):

$$r = k_2 q_e^2 \tag{11}$$

Fig. 7 shows plot of the kinetic data obtained for Purolite PFA 400 and A 500 resins. As shown in Fig. 7, removal of tartaric acid is fast. About 90% of tartaric acid was removed in 10 min. In 120 min more than 98% of tartaric acid removed from solution by gel and macroporous resins.

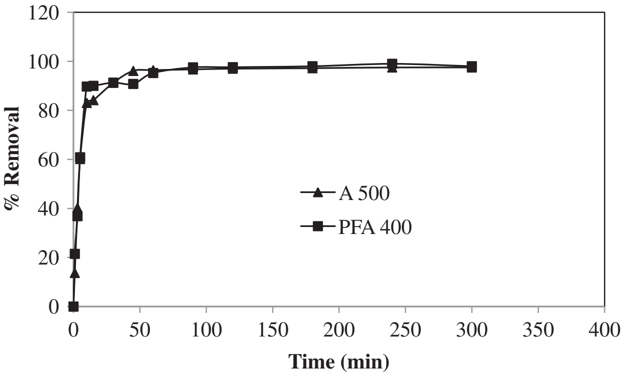

Fig. 7. Removal of tartaric acid vs. time.

As shown in Figs. 8 and 9; PFA 400 and A500 resins were fitted to sorption kinetics using the pseudo-first-order and pseudo-second-order kinetic models. When the linear correlation coefficients were compared, it was seen that sorption kinetics for PFA 400

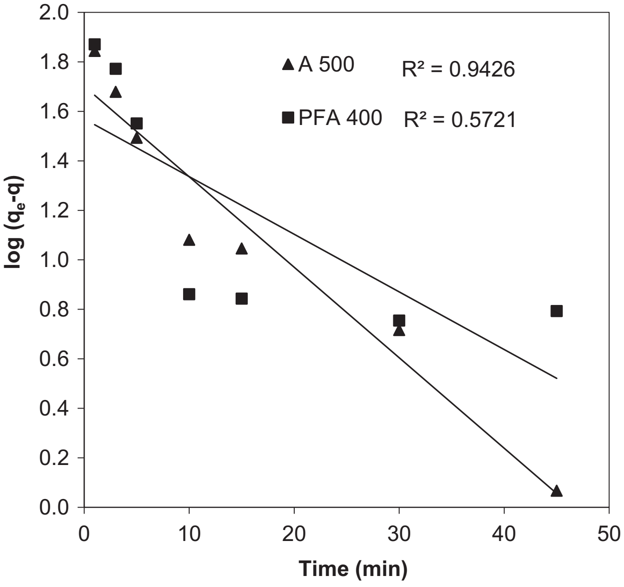

Fig. 8. Pseudo-first-order reaction kinetics for the adsorption of tartaric acid onto Purolite A500 and Purolite PFA 400.

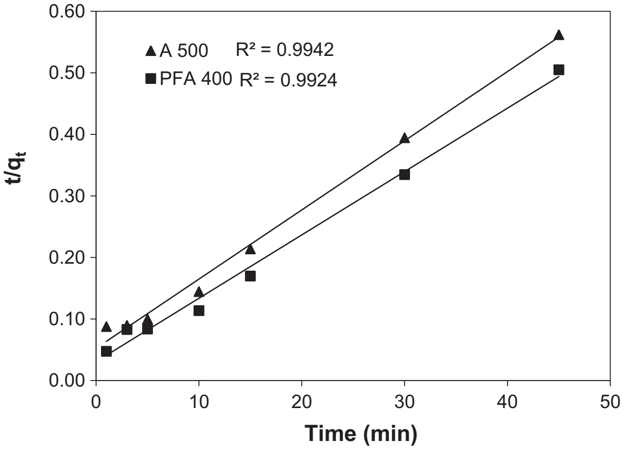

Fig. 9. Pseudo-second-order reaction kinetics for the adsorption of tartaric acid onto Purolite A500 and Purolite PFA 400.

Table 3
The calculated parameters of pseudo-first- and pseudo-second-order kinetic model

Kinetic model	Parameter	Purolite A500	Purolite PFA 400
Pseudo-first-order	k_1 (min^{-1})	0.0843	0.0537
	q_e (mg/g)	50.34	37.09
	R^2	0.94	0.57
Pseudo-second-order	k_2 (g/mg min)	2.39×10^{-3}	3.46×10^{-3}
	q_e (mg/g)	89.29	97.08
	r (mg/gmin)	19.08	32.57
	R^2	0.99	0.99

and A 500 resin agreed well to Ho pseudo-second-order mechanism.

The calculated parameters of pseudo-first- and second-order kinetic model were summarized in Table 3. As can be seen from Table 3 that k_2 and q_e values of gel resin (PFA 400) are higher than macroporous (A 500) resin.

3.6. Recovery of tartaric acid from wine fermentation vats water

The recovery of tartaric acid from wine wastes experiments were carried out with the samples taken from fermentation vats of Sevilen Wine factory (Turkey). In order to dissolve the precipitated CaT and KHT, the wine waste was acidified with HCl (pH 1.5). The dissociation reactions were shown in Eqs. (12) and (13).

$$\mathrm{CaT} + 2\mathrm{H_3O^+} \rightarrow \mathrm{H_2T} + \mathrm{Ca^{2+}} + 2\mathrm{H_2O} \qquad (12)$$

$$\mathrm{KHT} + \mathrm{H_3O^+} \rightarrow \mathrm{H_2T} + \mathrm{K^+} + \mathrm{H_2O} \qquad (13)$$

After dissolving the CaT and KHT, the solution was filtered to remove impurities. After filtration, $CaCl_2$ solution was added to filtrate and pH of solution was increased to 5–6 by adding NaOH. When the pH was increased only the CaT precipitated. The color of precipitate was not white; the other organic substance adsorbed and /or co-precipitated. After complete precipitation, the solution was filtrated by black label filter paper and CaT washed with pure water then CaT precipitate was transferred to warmer pure water (30°C). Purolite C 100 MBH cation exchange resins (H-form) were added to this solution and shaken for 24 h. All CaT was dissolved according to ion-exchange reaction between resin and dissolved Ca^{2+} ions (CaT dissolves in warm water) as shown in Eq. (14).

$$2\overline{R-H} + CaT \rightarrow H_2T + \overline{R_2Ca} \qquad (14)$$

The cation exchange resin was removed from solution. The color of solution was pale yellow. In order to remove organic impurities, some activated carbon was added to the solution and after 24 h shaking, the activated carbon was removed by filtration. The obtained colorless tartaric acid solution was evaporated and white tartaric acid crystalline was obtained with 90% recovery.

Similar recovery rates were obtained by other researchers. Recovery of tartaric acid from three industrial enological wastes by precipitation was carried out by Versari et al. [26]. The recovery of tartaric acid from eluate of anion exchange resin was 58%, from lees 83%, and 99.5 from creams of tartar. In another work; Matchett et al. [13] regenerated the tartrate-loaded anion exchange resin and 93% of tartaric acid was recovered from the effluent of anion exchange resins.

4. Conclusions

Removal and recovery of tartaric acid from aqueous solution and wine waste has been studied using strong base gel and macroporous resins over a range of pH conditions. The results show that pH is an important parameter on tartaric acid removal. When the pH is greater than 4; more than 98% of tartaric acid was removed by gel and macroporous resin. In acidic media (pH 2–3) the gel resin's removal performance is slightly better than macroporous resin. The calculated capacity of gel type resin is slightly higher than macroporous resin. The kinetics of tartaric acid adsorption on resins is very fast. In 10 min about 90% of tartaric acid was removed by gel type resin PFA 400 and 83% by macroporous A 500 resin. The hybrid precipitation-cation exchange resin system was used for recovery of tartaric acid from liquid wine waste. The obtained results showed that hybrid system can be used as an alternative method for the recovery of tartaric acid. The 90% of tartaric acid was recovered by hybrid system.

Acknowledgments

This study has been supported by The Scientific and Technological Research Council of Turkey [TUBITAK-Program code: 2209]. Authors would like to acknowledge Purolite Int. Co. for ion-exchange resins and Sevilen Wine factory for wine waste samples. They thank the Editor of this journal, the referees, and Mr Alparslan Sarıkaya for their kind suggestions to improve this paper.

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