

Effective Removal of Sulphates from Aqueous Solutions by Modified Orange Peel

Dalibor SUROVKA¹⁾, Eva PERTILE²⁾, Eliška SARČÁKOVÁ³⁾

¹⁾ Ing., Ph.D.; VŠB – TU Ostrava, Faculty of Mining and Geology, 17. listopadu 15, 708 33, Ostrava – Poruba, tel: 597 325 188; email: dalibor.surovka@vsb.cz

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Abstract

More recently, it has been shown that sulphate (SO_4) adsorption plays a role not only in mediating the effects of anthropogenic S emissions. It was found that SO_4 adsorption and desorption is important in buffering soil systems against extreme climatic events. For these reasons, correct understanding of SO_4 adsorption and desorption remains an important scope for geochemical research. Nowadays, several appropriate methods are available including cementation, chemical precipitation, membrane filtration, ion-exchange and and hydrogel. However, they often require a great investment, and generate large amounts of waste products. However, the adsorption methods are cheap methods, used on large scale. Technical and economic concerns have led to a focus on lost-cost adsorbents. Adsorption is commonly used and are very successful, environmentally friendly and acceptable economically in low concentrations contaminants.

The adsorption of sulphates from water solution using orange peel was studied by means of batch mechanism. This study addresses the important parameters of SO_4 with the aim of identifying optimal adsorption conditions. Many adsorption studies are available in the literature, which however focus on synthetic solutions. The aim of this study was to determine the dependency of sorption process on pH of the solution, the initial concentration of the sorbent, contact time and temperature. The Langmuir, Freundlich and Temkin isothermal models were used to describe the sorption isotherms of ions. They Langmuir model is more suitable for describing data than that of Freundlich. The adsorption efficiency of removing sulphates (q30 = 5 mg/g) by using orange peel was approximately 90%. When studying the kinetics it was discovered that the sorption process will follow the pseudo-second order. The thermodynamic parameters show exothermic character of sorption, and the processes will be spontaneous and favourable. The results indicate that it is possible to use orange peel effectively for removing sulphates and selected metals from waste water.

Keywords: sorption, sulphates, sorption kinetics, equilibrium isotherms, thermodynamics, kinetic rate equations

Introduction

Many industrial activities discharge sulfateand metal-containing wastewaters, including the manufacture of pulp and paper, mining and mineral processing, and petrochemical industries. In order to reuse the water in production processes, to comply with environmental regulations, and to recover metals, treatment of these wastewaters is required. (Shehnaaz Moosa et al., 2005)

The mobility of anions in the aquatic environment is typically regulated by adsorption at the solid/water interface and by competition among various anion species for surface binding sites. The magnitude of these competitive interactions must be determined to predict and model anion migration. (C.H. Wu et al., 2002). Most of the studies on ion removal from wastewater in the mining and metallurgical fields are concerned with cations removal, namely Cu, Zn, Cd, Hg, Pb etc. Yet, high concentrations of anions such as phosphate, nitrate, vanadate, molybdate, thiocyanaty, chromate and sulfate ions are present in the mining effluents and discharged without any treatment. Sul-

fate is generated from dissolution of sulfate bearing minerals, chemical and biological oxidation of sulfide ores like pyrite and from paper, paint, plastic and rubber industries (C. Namasivayam & D. Sangeetha 2008). Therefore, removal of the anion ground water is of great importance for the environment. Aim of this study was to evaluate the potential of orange peel as an active sorbent to remove sulfates from water. Was also examined adsorption dynamics, equilibrium studies and the effects of pH and temperature.

Materials and Methods Adsorbent

Orange peel used in this study were obtained from various sources. Orange waste was first shredded into smaller pieces, washed several times with distilled water to remove dirt, sugars, tannins and pigments. Waste is dried at $60\pm1^{\circ}\text{C}$ 48 h. to constant weight. Washing and drying the crushed material of the size of 0.5 to 1 mm. This adjusted orange peel was activated in 1 M HCl and 1 M NaOH for 30 minutes. After the activation

²⁾ Doc. Mgr., Ph.D.; VŠB – TU Ostrava, Faculty of Mining and Geology, 17. listopadu 15, 708 33, Ostrava – Poruba, tel: 597 325 188; email: eva.pertile@vsb.cz

³⁾ Ing.; VŠB – TU Ostrava, Faculty of Mining and Geology, 17. listopadu 15, 708 33, Ostrava – Poruba, tel: 597 325 188; email: eliska.sarcakova@vsb.cz

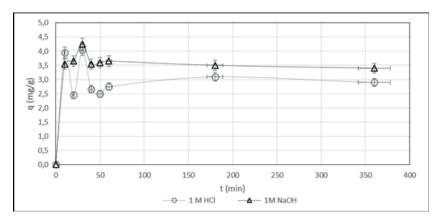


Fig. 1. Effect of contact time removing SO_4^{2-} on chemically modified orange peel (cs = 20 g/l, ci = 100 mg/l, stirring speed of 150 rev./min, t = 25 ± 2 °C, without pH adjustment)

Rys. 1. Wpływ czasu na usunięcie $SO_4^{\ 2^-}$ na modyfikowanej chemicznie skórce pomarańczowej (cs = 20 g/l, ci = 100 mg/l, prędkość mieszania 150 obr/min, t = 25 ± 2°C, bez regulacji pH)

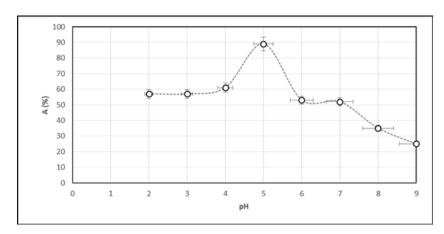


Fig. 2. Effect of pH on removal SO_4^{2-} (cs = 20 g/l, ci = 100 mg/l, stirring speed of 150 rev./min, t = 25 ± 2°C, the exposure time 30 min)

Rys. 2. Wpływ pH na usuwanie SO_4^{2-} (cs = 20 g/l, ci = 100 mg/l, prędkość mieszania 150 obr/min, t = 25 ± 2°C, czas 30 min)

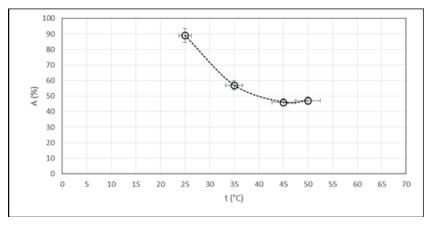


Fig. 3. Effect of temperature on removal SO_4^{2-} (cs = 20 g/l, ci = 100 mg/l, stirring speed of 150 rev./min, $t = 25 \pm 2^{\circ}C$, the exposure time 30 min)

Rys. 3. Wpływ temperatury na usuwanie SO_4^{2-} (cs = 20 g/l, ci = 100 mg/l, prędkośc mieszania 150 obr/min, t = 25 ± 2°C, czas 30 min)

process, the adsorbents and then filtered, washed with bidistilled water till being neutral, and then dried at 60±1°C 48 h

Chemicals

Sulfates stock solution (100 mg/L) was prepared by dissolving K₂SO₄ (PENTA) in distilled water and then added to the mark to the desired concentration. Conditioning agent was prepared from HCl, NaCl, glycerol, ethanol and distilled water. All chemicals were used in the purity p.a.

Analytical method

In the adsorption study, the conditions examined sorption SO₄²⁻ in the orange peel, and the conditions for maximum adsorption. Of analyzing the solution was used UV-VIS spectrophotometer HACH DR2800 at a wavelength of 420 nm. Each model sample 50 ml (100 mg/L) was in contact with 1 g of sorbent of different length at the orbital shaker IKA KS 4000i control at constant speed (150 rpm). At pre-determined time interval, the suspension was filtered through filter paper PRA-GOPOR with a density of 84 g/m² (fa Pragochema) and the filtrate was analyzed to evaluate the concentration of SO₄²⁻ in the sample. After evaluating sorption is further investigated and improved the conditions by adjusting pH and increasing temperature. The analysis was made according to EPA methods 375.4. Removal efficiency SO₄²⁻ was recalculated (1) as:

$$q = \left(c_i - c_f\right) \cdot \frac{V}{S} \tag{1}$$

where: ci is the initial concentration (mg/l), cf is the measured concentration (mg/L), q is the amount of metal adsorbed to the adsorbent (mg/L), V is the amount of solution (ml), S the weight of the sorbent (Haghsheno R., 2009).

Results and Discussion Kinetic Studies

Kinetic studies were carried out by performing absorption experiments the initial metal concentration 100 mg/L (50 ml solution) for different times (10–360 min) to an orange peel with the size of 0.5 to 1 mm. Studied peel was activated in NaOH and HCl, concentracion 1 mol/L, activated 30 min.

As shown in fig. 1 in the first 30 minutes of the best and fast sorption uptake is achieved with a time of 30 min. And then followed by desorption equilibrium of 60 minutes. For further experiments was chosen adsorbent activated in 1 M NaOH and the activated time was 30 minutes.

Effect of pH

One of the most important factors on which depend biosorption process, the pH value, witch greatly affects the dissolution and behavior of metals in solution and also affects the activity of functional groups on the surface of the cell wall selected biosorbents. The experiment was made with 1 g orange peel at different pH (2–10). Each volume pH of model metal solutions is maintained using buffers.

As seen in Fig. 2, the increase of pH from 2 to 5 with activated orange peel sodium hydroxide at a concentration of 1.0 mol/L after 30 min period, the end of exposure 30 min observed percentage increase in the removal of sulphate from 57% to 90%. With further increase of the pH value sulphate removal efficiency decreased. The increase in the adsorption of sulphate may be explained by the increase of protons in an acid medium.

Effect of temperature

Temperature affects the biosorption process in most cases less compared to the pH. Effect of temperature was studied at 25, 35, 45, 50°C \pm 1°C initial sulfate concentration was 100 mg/L (50 ml solution), weighed 1,000 0 g sorbent. Figure 3 shows the effect of temperature on removal SO₄²⁻.

Fig. 3 describes the effect of temperature on the process of removing SO_4^{2-} . When using chemically modified orange peel reached sorption maxima at $25\pm2^{\circ}$ C, which was the removal of 89% (q30 = 4.45 mg/g) sulphates. The reduction of sulphate removal at high temperatures is probably due to damage of active binding sites on the adsorbent, and the increasing tendency of desorption sulfate ions from the solid to the solution interface.

Adsorption isotherms

In general, the adsorption isotherm is a curve that describes the phenomenon governing the delay or the mobility of a substance from a porous material into the aquatic environment at constant temperature and pH (G. Limousin et al., 2007; S.J. Allen et al., 2004). Isotherms are typically mathematical relationship, which play a vital role in the modeling analysis, the graph structure and usable adsorption systems. They are usually represented graphically and expresses the relationship solid phase prior to its residual concentration (M.C. Ncibi, 2008).

Langmuir I. isotherm

Langmuir adsorption isotherm, was originally developed for the adsorption described gas-solid

Tab. 1. List of equations used adsorption isotherms

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Isotherm	Non-linear form	Linear form	Plot	Reference	
Langmuir I.	$q = \frac{q \cdot b \cdot C}{1 + b \cdot C}$	$\frac{C}{q} \frac{1}{q} \cdot C + \frac{1}{q \cdot b}$	$\frac{C}{q} v sC$	(Langmuir, 1916)	
Freundlich	$q K \cdot C^{\frac{1}{n}}$	$\ln(\mathbf{q}) = \ln K + \frac{1}{n} \ln(C)$	ln⊠q v sln(C)	(Freundlich, 1906)	
Temkin	q b.ln(K.C)	$q b \cdot \ln \boxtimes K \boxtimes + b \cdot \ln (\bigcirc C)$	q v sln(C)	(Behnamfard, 2009)	

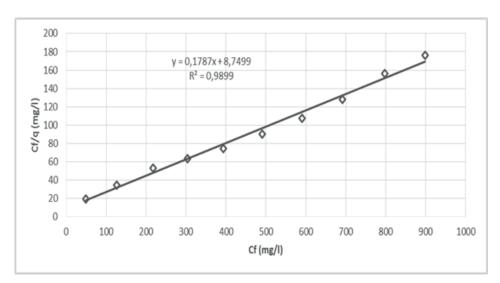


Fig. 4. Langmuir isotherm dependence C_f/q vs. C_f (cs = 20 g/l, stirring speed of 150 rev./min, t = 25 ± 2°C, the exposure time 60 min, pH 5.0)

Rys. 4. Izoterma Langmuira , zależność $C_{\rm f}/q$ od $C_{\rm f}$ (cs = 20 g/l, prędkość mieszania 150 obr/min, t = 25 ± 2°C, czas 60 min, pH 5.0)

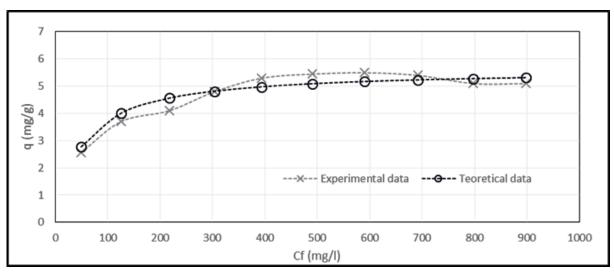


Fig. 5. Non-linear Langmuir isotherm dependence C_f vs q (cs = 20 g/l, stirring speed of 150 rev./min, t = 25 ± 2°C, the exposure time 60 min, pH 5.0

Rys. 5. Nieliniowa izoterma Langmuira – zależność C_f od q (cs = 20 g/l, czas mieszania 150 obr/min, t = 25 \pm 2°C, czas = 60 min, pH 5.0

phase on activated carbon. Traditionally used to quantify the performance of a diversity of organic adsorbents (I. Langmuir, 1916). In his formulation, the empirical model assumes monolayer adsorption. In linear form (2) it can be written:

$$\frac{C_f}{q} = \frac{1}{q_{\text{max}}} \cdot C_f + \frac{1}{q_{\text{max}} \cdot b}$$
 (2)

where: q (mg/g) and C_f (mg/l), is the amount of adsorbed SO_4^{2-} per unit weight of sorbent, q_{max} is the maximum adsorption SO_4^{2-} per unit weight of the binder, which forms a complete monolayer on the surface of, b is a constant related to the affinity of binding sites (L/mg) (K. Vijayaraghavan, 2006). Fig. 4 is a graphical representation (C_f /q vs. C_f) Langmuir I. isotherms for the adsorption of SO_4^{2-} on orange peel.

Langmuir isotherm is generally linearized five ways, all of which are looking for, the one that best fits the experimental date. The correlation coefficient R² indicates the high compliance linearized model and measured results. Measured and calculated values are given in Table 2.

Non-Linear Langmuir isotherm

From the linear dependence of C_f/q vs C_f to be determined constants of linearized forms of the adsorption isotherms, which are used in the construction of the non-linear dependence of the equilibrium adsorption capacity q of the equilibrium concentration of sulfate in the solution of C_f (Figure 5). Nonlinear dependence describes the relationship between measured and calculated data of adsorption capacity.

The essential characteristics of Langmuir isotherm can be explained in terms of dimensionless constant separation factor (R_L) which is expressed as (3) (Kadirvelu et al., 2001):

$$R_L = \frac{1}{1 + bc_i} \tag{3}$$

where: c_i and b has already been explained above. Thus, constant separation factor (R_L) is a positive number whose magnitude determines the feasibility of the adsorption process. The R_L values for each of the different initial concentrations used are between 0 and 1 indicating favorable adsorption of sulfates onto differently modified orange peel.

Freundlich isotherm

Freundlich isotherm is rather well-known formula for the description of non-ideal and reversible adsorption is not limited to the creation of monolayer adsorption. The empirical model can be applied to multi-layer adsorption, the uneven distribution of heat and adsorption affinity for the uneven surface (A.W. Adamson & A.P. Gast, 1997). Linearized form (4) of the equation can be written in the form (Behnamfard A. & Salarirad M.M., 2009)

$$\ln(q) = \ln K_f + \frac{1}{n} \cdot \ln(C_f)$$
 (4)

where: q is the amount of absorbed substances (mg/g), K_f is the Freundlich constant (mg/g)(L/mg)1/n, Cf is the final concentration of sorbate in solution (mg/L), and n is a constant intensity of adsorption. Currently, the Freundlich isotherm is widely used in heterogeneous systems, in particular for organic compounds, or the adsorption of substances highly interactive charcoal and molecular sieves. Figure 6 shows a graphic representation of the curve $(\ln{(C_f)})$ vs. $\ln{(q)}$) uptake SO_4^{2-} on the orange peel.

Graph Freundlich isotherm was constructed from the values of $\ln q$ and C_f . Has the linearized shape and the value of R^2 is clear that the model does not describe the data Freundlich more suitably than Langmuir equation (Treybal, 1981). The measured results and the calculated values are in Table 2.

Non-Linear Freundlich isotherm

The Freundlich isotherm as a two-parameter model is the earliest known relationship describing the sorption equation. The model applies to sorption onto heterogeneous surfaces with a uniform energy distribution. The application of the Freundlich equation (5) suggests that sorption energy exponentially decreases on completion of the sorption centres of the sorbent (Freundlich, 1906). The model can be expressed as:

$$q = K_f \cdot C_f^{\frac{1}{n}} \tag{5}$$

where: q is the amount of absorbed substances (mg/g), C_f is the concentration of SMET at sorption equilibrium is the final concentration of sorbate in solution (mg/L), K_f is the constant indicative of the relative sorption capacity of the sorbent ((mg/g)(L/mg)1/n), and 1/n is the heterogeneity factor.

Obviously, the Langmuir equation provided more accuracy in prediction than the Freundlich model in comparison with the result fitted by the split line model. The Freundlich model assumed no limited level of sorption (saturation) value, and thus, it was inappropriate to model the case stud-

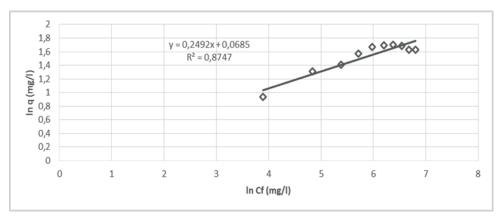


Fig. 6. Graphical representation of the linearized Freundlich isotherm (cs = 20 g/l, stirring speed of 150 rev./min, $t = 25 \pm 2$ °C, the exposure time 60 min, pH 5.0)

Fig. 6. Graphical representation of the non-linearized Freundlich isotherm (cs = 20 g/l, stirring speed of 150 rev./min, $t = 25 \pm 2$ °C, the exposure time 60 min, pH 5.0)

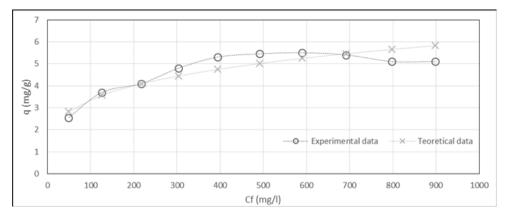


Fig. 7. Graphical representation of the non-linearized Freundlich isotherm (cs = 20 g/l, stirring speed of 150 rev./min, $t = 25 \pm 2$ °C, the exposure time 60 min, pH 5.0)

Rys. 7. Ilustracja graficzna nieliniowej izotermy Freundlicha (cs = 20 g/l, prędkość mieszania 150 obr/min, $t = 25 \pm 2^{\circ}\text{C}$, czas 60 min, pH 5.0)

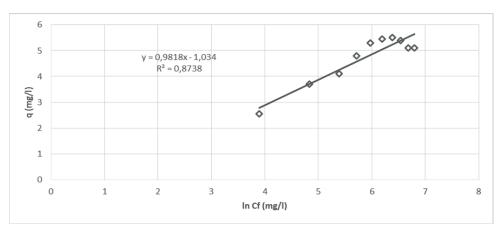


Fig. 8. Graphical representation of the linearized Temkin isotherms (cs = 20 g/l, stirring speed of 150 rev./min, $t = 25 \pm 2$ °C, the exposure time 60 min, pH 5.0)

Rys. 8. Graficzna ilustracja linearyzowanej izotermy Temkina i (cs = 20 g/l, prędkośc mieszana 150 obr/min, $t = 25 \pm 2^{\circ}\text{C}$, czas 60 min, pH 5.0)

Tab. 2. Measured	and calculated	licotherm	constants
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Isotherm	Constant		Correlation coefficient	Separation factor	Measured q	Theoretical q
	b (L/mg)		\mathbb{R}^2	RL	q (mg/l)	q _{max} (mg/l)
Langmuir	0,02		0,99	0,33	5,5	5,96
	n	$\mathbf{K_f}$ $(mg/g)(L/mg)^{1/n}$				
Freundlich	4,01	1,07	0,87			
	bt	$K_t(L/g)$				
Temkin	0,98	0,35	0,87			

ied by Özacar (2003) with the typical saturated value. It was found that the Freundlich curve deviated from experimental data, particularly in the transition region, and this resulted in an inexact estimation (Fig. 7).

Temkin isotherm

Temkin the initial isotherm model describing the adsorption of hydrogen on platinum electrodes in acidic solutions. Isotherm contains a factor that literally takes into account the interaction of adsorbent - adsorbed. Shape equations in linearized form (6) is (A.W. Adamson, A.P. Gast, 1997):

(6)

 $q = b_t \cdot \ln(K_T) + b_t \cdot \ln(C_f)$ where: q is the amount of absorbed substances (mg/g), b_t is the maximum under the conditions q (mg/g), K_T Temkin the equilibrium constant (L/mg) and C_f is the final concentration of sorbate in solution (mg/L). Ignoring extremely low and high levels of concentration, the model assumes that the heat of adsorption of molecules in the layer would be reduced to a more linear logarithm coverage (C. Aharoni & M. Ungarish, 1977). Figure 8 is a graphical representation (ln C_f vs q) Temkin sorption isotherm.

Although the correlation coefficient of linear regression model by Temkin R² indicate a similar pattern function, it is interesting the same course with logarithmic regression of experimentally de-

rived values. Temkin isotherm shape is sort of a combination between Langmuir and Freundlich model. The obtained parameter values are given in Table 2.

Conclusion

This study shows that the orange peel can be used as a low-cost adsorbent for the removal of sulfate ions. pH is a key variable to remove sulfates. In this context, reaching the maximum of removing sulfates around pH 5 Temperature had a negative impact.

The correlation coefficient R² indicates the high compliance linearized model with measured results, and are in good agreement with Langmuir I. isotherm, from which the maximum balance evaluated as 5.959 mg/L (90%). It should be noted that it may be absorbed quite a large number of sulfate by a relatively small amount of orange peel (1g orange peel for removal of 90% solution of the model solution of 100 mg/L). Easy accessibility of the adsorbent, high adsorption capacity, and most importantly, the operation of this system is expected, that there is no dangerous sludge and by reducing operating costs, making this method of removal is an attractive and competitive.

Acknowledgment

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Usuwanie siarczanów z roztworów wodnych przy użyciu zmodyfikowanej skórki pomarańczowej Badania wykazały, że adsorpcja siarczanów ($\mathrm{SO_4}$) ma kluczową rolę nie tylko w analizie wpływu antropogenicznej emisji S. Odkryto, że adsorpcja $\mathrm{SO_4}$ i desorpcja są ważne w instalacjach buforowania gleb przed ekstremalnymi wydarzeniami klimatycznymi. Z tych powodów, szersze rozumienie adsorpcji $\mathrm{SO_4}$ i desoprcji pozostaje ważnym tematem badań geochemicznych. Obecnie dostępnych jest kilka metod oczyszcznia wód – wliczając w to cementację, strącanie chemiczne, filtracje membranową, wymianę jonową oraz hydrożel. Jednakże, wymienione metody badań wymagają sporych inwestycji i wytwarzają ogromne ilości odpadów, podczas gdy same metody adsorpcyjne są tanie i używane na szeroką skalę. Okoliczności ekonomiczne oraz techniczne doprowadziły do skupienia się na niskokosztowych adsorbentach. Adsorpcja jest powszechnie stosowana z satysfakcjonującymi wynikami, jest przyjazna środowisku oraz ekonomiczne akceptowalna przy niskich stężeniach kontaminatu.

Zbadano adsorpcję siarczanów z roztworu wodnego z użyciem skórki pomarańczowej. Badanie miało na celu zbadanie istotnych parametrów adsorpcji SO₄ ze szczególnym dążeniem do identyfikacji optymalnych warunków adsorpcji. Literatura przedstawia wiele badań nad adsorpcją, jednakże jedynie z wykorzystaniem roztworów syntetycznych. Celem niniejszych badań było wyznaczenie zależności procesów sorpcyjnych od pH roztworu, stężenia początkowego sorbentu, czasu kontaktu oraz temperatury. Aby opisać izotermę sorpcji jonów użyto modeli izotermicznych Langmuir'a, Freundlich'a i Temkin'a. Model Langmuir'a jest wygodniejszy do opisu danych niż model Freundlich'a. Skuteczność adsorpcji w usuwaniu siarczanów (q30 = 5mg/g) przy użyciu skórki pomarańczy wyniosła około 90%. Podczas badania kinetyki odkryto, że procesy sorpcji podlegają reakcji pseudo-drugiego rzędu. Parametry termodynamiczne pokazały właściwości egzotermiczne sorpcji, a procesy zachodzą spontanicznie i korzystnie. Wyniki potwierdziły, że można skutecznie wykorzystywać skórkę pomarańczy do usuwania siarczanów i wybranych metali ze ścieków wodnych.

Słowa kluczowe: sorpcja, siarczany, kinetyka sorpcji, równanie izotermy, termodynamika, współczynnik kinetyczny