ADSORPTION OF SELENITE ION FROM AQUEOUS SOLUTION BY SOLID PINEAPPPLE WASTE

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To my beloved husband, family and friends

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ABSTRACT

In the pineapple canning industry, almost forty percent of pineapple portion is rejected during the production process. Most of the rejected portion is used as ruminant and for alcohol production. In the present study, the untreated solid pineapple waste (SPW) was used to adsorb selenite ion (Se(IV)) from aqueous solution. The Se(IV) loaded and unloaded SPW were characterized using proximate analysis, atomic absorption spectrometry, scanning electron microscopy, energy-dispersive X-ray spectroscopy, thermogravimetry analysis, differential scanning calorimetry and Fourier transform infrared spectroscopy. Various parameters that affect the adsorption such as initial pH, contact time, temperature, mass of adsorbent and concentration of adsorbate were investigated to determine the optimum adsorption conditions. Kinetics of the adsorption was investigated by pseudo first-order and pseudo second-order models. Equilibrium studies were carried out by Langmuir, Freundlich and Temkin isotherms. Thermodynamic studies of the adsorption were performed using van't Hoff equation. Desorption studies of the Se(IV) loaded SPW were carried out under various conditions such as initial pH, desorption time, ratio mass of loaded SPW to reaction volume and concentrations of humic acid and competing ions such as chloride, phosphate and sulphate ions. Under the optimum adsorption conditions the Se(IV) content of SPW could be increased by 100 folds from less than 10 μg/g to nearly 1000 μg/g. The adsorption optimum conditions were at pH 6, adsorption duration t = 60 min, initial concentration of Se(IV) aqueous solution C_0 = 10 mg/L, SPW weight was 3 g in 50 mL of reaction volume and temperature was 80°C. The adsorption rate of Se(IV) by SPW under the optimum conditions fitted the pseudo second order with rate constant, k_2 = 4.8539 mg/g min while the adsorption of Se(IV) by SPW best fitted Temkin isotherm (R^2 = 0.9963), followed by Langmuir isotherm (R^2 = 0.9830) and the Freundlich isotherm (R^2 = 0.9702), which indicated the dominance of chemisorption. The van't Hoff equation applied to this adsorption indicated that the adsorption process is endothermic, favourable and spontaneous. The adsorbed Se(IV) can be leached out gradually under the influences of pH, ratio of mass of loaded SPW to reaction volume and the presence of humic acid, phosphate, sulphate and chloride ions.

ABSTRAK

Dalam industri pengetinan nenas, hampir empat puluh peratus daripada bahagian nenas lazimnya disingkirkan semasa proses penghasilan produk. Kebanyakan daripada bahagian yang disingkirkan itu dijadikan sebagai sumber makanan haiwan dan untuk penghasilan alkohol. Dalam kajian ini, sisa pepejal nenas (SPW) yang tidak mengalami sebarang proses rawatan telah digunakan untuk menjerap ion selenit (Se(IV)) daripada larutan akueus. Kajian mengenai SPW yang tidak dijerap dan telah dijerapkan dengan Se(IV) telah dijalankan dengan mengunakan analisis proksimat, spektrometri serapan atom, mikroskopi pengimbas elektron, mikroskopi serakan tenaga sinar-X, analisis termogravimetri, kalori pengimbas kebezaan dan spektroskopi inframerah transformasi Fourier. Pelbagai parameter seperti nilai awal pH, masa interaksi, suhu, berat bahan penjerap dan kepekatan bahan yang dijerap telah dikaji untuk menentukan keadaan jerapan yang optimum. Kajian mengenai kinetik jerapan ini telah dijalankan melalui model tertib pseudo pertama dan tertib pseudo kedua. Kajian keseimbangan pula telah dijalankan menggunakan isoterma Langmuir, Freundlich and Temkin. Kajian termodinamik dalam proses jerapan ini telah dijalankan dengan menggunakan persamaan van't Hoff. Kajian penyahjerapan daripada SPW yang telah dijerapkan dengan Se(IV) telah dijalankan untuk mengkaji potensi pembebasan Se(IV) berdasarkan beberapa parameter seperti nilai awal pH, masa interaksi, nisbah jisim SPW terisi kepada isipadu tindak balas dan kepekatan asid humik dan ion persaingan seperti klorida, fosfat dan sulfat. Pada keadaan jerapan optimum, kandungan Se(IV) dalam SPW boleh meningkat daripada 10 μg/g kepada 1000 μg/g. Keadaan jerapan yang optimum ialah pada pH = 6, masa jerapan selama 60 min, kepekatan awal iaitu 10 mg/L, berat SPW iaitu 3 g dalam 50 mL isipadu tindak balas dan suhu pada 80°C. Kadar jerapan Se(IV) oleh SPW bersesuaian dengan model tertib pseudo kedua dengan pemalar kadar, k_2 = 4.8539 mg/g min. Jerapan ion Se(IV) menggunakan SPW yang memperoleh padanan yang terbaik ialah melalui isoterma Temkin (R^2 = 0.9963), diikuti oleh isoterma Langmuir (R^2 = 0.9830) dan isoterma Freundlich (R^2 = 0.9702), di mana proses kimia telah mendominasi proses jerapan ini. Persamaan van't Hoff yang telah diaplikasikan dalam kajian ini menunjukkan bahawa proses jerapan yang berlaku ialah melalui proses endotermik, memuaskan dan berlaku secara spontan. Kandungan Se(IV) yang telah dijerap boleh dilarutlesap secara beransur-ansur di bawah pengaruh pH, nisbah jisim SPW terisi kepada isipadu tindak balas dan kehadiran asid humik, ion fosfat, sulfat dan klorida.

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LIST OF ABBREVIATIONS

C	-	Carbon
C_0	-	Initial concentration
CO	-	Carbon monoxide
CO_2	-	Carbon dioxide
g	-	Gram
kg	-	Kilogram
L	-	Liter
M	-	Molar
mg	-	Milligram
min	-	Minute
mL	-	Milliliter
µg	-	Microgram
Se	-	Selenium
S/L	-	Solid to liquid ratio
t	-	Time
T	-	Temperature
w	-	Weight
$\Delta G°$	-	Gibbs free energy
$\Delta H°$	-	Enthalpy change
$\Delta S°$	-	Entropy change

CHAPTER 1

INTRODUCTION

1.1 Background of Study

According to Malaysian Pineapple Industry Board, Malaysia produced about 156,000 metric tons of pineapples and the quantity of solid pineapple waste yield was estimated about 48,000 tons from pineapple processing factories (Goh *et al.*, 2010). During the canning process, about 40% to 80% of the discarded portion considered waste consist of crown, the outer peel and the central core of the pineapple (Koffi and Han, 1990). Solid pineapple waste contains carbohydrate, simple sugars, cellulose, hemicellulose and lignin (Koffi and Han, 1990;Ferri et al., 2003;Tuzen and Sari, 2010). Released components into the waste streams and finally into the environment causes serious pollution problems due to their high biochemical oxygen demand (BOD) and chemical oxygen demand (COD). Hence, proper treatment and disposal of these released components are important part of the pineapple industry. In practice, most of pineapple processing waste is either fermented for alcohol or organic acids such as citric acid production, vinegar and wine, and used as ruminants (Tran, 2006).

Concern over the abundance of the pineapple waste and possible impacts on the environmental has raised interest in utilization of solid pineapple waste for various applications. In recent years, number of reports indicates the increasing interest on the use of agriculture waste as adsorbent for removal toxic materials such as metals from wastewaters. Agriculture waste offers high efficiency metal ions uptake due to its unique chemical composition for metals binding capability and possibility of metal recovery. Utilization of biomass, known as biosorption for removal of metal from wastewater is driven by the interests in connection to health effect of metal toxicity, the economics of metal recovery and scientific studies to understand the metal adsorption behavior (Volesky, 2007). In this respect, solid pineapple waste has an interesting potential for selenium sorption to remove selenium from wastewater, as well as a means for enrichment to agriculture soil with selenium deficiency problems. Recent published papers have demonstrated outstanding selenium anions uptake capability by agricultural waste in terms of efficiency, rapidity, low cost, flexibility, simplicity of design and ease of operation (Rafatullah *et al.*, 2010). A wide variety of potential adsorbents to remove selenium anions have been prepared from agriculture waste either in natural or after physical or chemical modification form such as sugarcane bagasse, wheat bran, green algae, peanut shell and rice husk (Kailas et al., 2009;Hasan et al., 2010;Tuzen and Sari, 2010;El-Shafey, 2007a;El-Shafey, 2007b) .

Selenium is an interesting element due to its contradicting role to human health depending on concentration. Selenium is an essential micronutrient to human health and as an antioxidant. At high concentration it is toxic to human health but lack of this element will create selenium deficiency problems in the form of diseases. The recommended daily intake of this element is from 50 to 220 μg/day but it is toxic only at twice those required, which is above 400 μg/day (Tuzen and San, 2009).

Industrially, selenium is a by-product of certain process such as electrolytic copper refining and combustion of fossil fuels. It is used in manufacture of glass, alloys, toner of photographic prints and electronics such as photocopying, photocells, light meters, solar cells, rectifiers, and xerography equipments. Selenium concentration in industrial effluent may vary from 1 to 7000 μg/L (Kapoor *et al.*, 1995). According to the Ministry of Natural Resource and Environmental (NRE), about 13 from 30 local industrial factories produced heavy metal sludge including selenium. Hence, it is possible that selenium will end up polluting the environment by the discharge of wastewater into the drainage system. According to Malaysia Environmental Quality (Industrial Effluents) regulations, the maximum acceptable concentration of selenium discharge in industrial effluent is 0.02 mg/L, whereas according to Ministry of Health Malaysia, the national standard for drinking water quality for selenium is 0.01 mg/L. There are several ways to remove selenium anions from contaminated wastewater including chemical precipitation, coagulation, ion exchangers, chemical oxidation and reduction, reverse osmosis, electro dialysis, ultra filtration and activated carbon adsorption. However, these conventional treatments contribute to limitation include less efficiency, high in cost, sensitive operating conditions and production of secondary sludge (Sud *et al.*, 2008).

1.2 Problem Statement

There has been increasing interest to use plant biomass or agriculture waste such as rice husk, peanut shell, sugarcane bagasse, wheat bran and green algae to adsorb Se(IV) from aqueous solution. Information on the adsorption of Se(IV) by solid pineapple waste is important because of potential used in Se(IV) toxicity removal as well as Se(IV) enrichment of SPW which can be further use to soil enhancement for Se(IV) deficiency problem. It is important to investigate the Se(IV) adsorption by solid pineapple waste with respect to initial pH, contact time, temperature, dosage of adsorbent and adsorbate concentration. It is necessary to understand the kinetics of adsorption which reflects the rate of metal ions uptake by the adsorbent. Meanwhile for equilibrium of the adsorption can be investigated by fitting approach to Langmuir, Freundlich and Temkin isotherms. The van't Hoff equation normally used to determine the thermodynamic parameters of adsorption process. Considering the potential of the adsorbed Se(IV) to be slowly released for soil enhancement, it is important to investigate the leachability with respect to pH, contact time, solid to liquid ratio, concentration of competing ions and humic acid.

1.3 Objectives of the Research

This research was carried out to study the adsorption of selenite ion from aqueous solution. The objectives of this study are as follows:

i. To investigate the general characteristics of the solid pineapple waste.

ii. To investigate the effect of initial pH, contact time, temperature, adsorbent dosage and adsorbate concentration on the Se(IV) adsorption by solid pineapple waste.

iii. To investigate the equilibrium behavior of Se(IV) adsorption by solid pineapple waste based on Langmuir, Freundlich and Temkin isotherms.

iv. To investigate the kinetics of Se(IV) adsorption by solid pineapple waste based on pseudo first-order and pseudo second-order models.

v. To investigate the leachability of the Se(IV) adsorbed by solid pineapple waste under various leaching conditions such as initial pH, contact time, solid to liquid ratio, competitive anions and humic acid.

1.4 Significance of Study

The adsorption study of Se(IV) by an agriculture waste such as solid pineapple waste has a wide implication including understanding the kinetic, equilibrium and thermodynamic of the process. The study also provides information on the prospect of utilizing solid pineapple waste as a biomass based material selenium enrichment of the agriculture soil for enhancement crop production quality. Reduction of environmental pollution due to the solid pineapple waste may be achieved by converting them into value added products. Since selenium has a dual role as an essential micronutrient in diet at small amounts and toxicity at high concentration, the present study represents an approach probably most suitable to fulfill both the needs for selenium toxicity removal from wastewaters and selenium enrichment method for agriculture soil enhancement and supplementation. Therefore, the selenium adsorption and desorption batch experiments were conducted to investigate the sorption behavior and the suitability of the adsorbent within various environmental effects, kinetics, equilibrium and thermodynamic studies.

1.5 Scope of the Research

This research involves utilization of solid pineapple waste prepared from discarded components from pineapple canning process. Proximate analysis was employed to investigate the physico-chemical properties of the solid pineapple waste. The morphological properties and the nature functional groups analysis were investigated using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDAX) and Fourier transform infrared spectroscopy (FTIR). The thermal analysis of both blank and Se(IV) loaded solid pineapple waste were investigated by thermogravimetry analysis (TGA) and differential scanning calorimetry (DSC).

Batch adsorption experiments of Se(IV) were conducted to determine the effect of various parameters such as initial pH, contact time, temperature, adsorbent dosage and adsorbate concentration. The results obtained were analyzed using intra-particle diffusion model, kinetic models such as pseudo first-order and second-order, equilibrium isotherms analysis using Freundlich, Langmuir and Temkin isotherms, and thermodynamic parameters using van't Hoff equation to determine the Se(IV) adsorption behavior by solid pineapple waste. Desorption batch experiments were carried out to determine the effect of Se(IV) leachability from adsorbed solid pineapple waste as functions of pH, contact time, solid to liquid ratio, concentration of competiting ions and humic acid.

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