# ADSORPTIVE REMOVAL OF PHOSPHATE FROM AQUEOUS SOLUTION USING SPONGE GOURD FIBRE

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PERPUSTAKAAN IINIVERSITI MALAYSIA SABAH

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#### ABSTRACT

The removal of phosphate ion from aqueous solution by natural sponge gourd fibre was investigated at different contact time and initial phosphate concentration according to batch method. As comparison, a similar study was carried out using pretreated sponge gourd fibre. The final phosphate concentration was determined according to Ascorbic Acid method. The results showed that the adsorption process for both natural and treated sponge gourd fibre was rapid during the first 10 minutes. Equilibrium, however, was attained after 60 minutes. The adsorption fitted well to pseudo second order kinetic model. Phosphate adsorption by natural sponge gourd fibre fitted well to Freundlich isotherm while the pre-treated sponge gourd fitted well to Langmuir isotherm. The maximum adsorption capacity of natural and treated sponge gourd fibre was  $87.7 \mu g/g$  and  $2500 \mu g/g$ , respectively. Overall, pre-treatment of the sponge gourd fiber increased its phosphate adsorption efficiency.



# PENYINGKIRAN JERAPAN ION FOSFAT DARI LARUTAN AKUA DENGAN SERABUT PETOLA

### ABSTRAK

Penyingkiran ion fosfat dari larutan akua oleh serabut petola semula jadi telah dikaji mengikut keadah kelpmpok pada keadaan masa dan kepekatan awal yang berbeza. Kajian yang sama dilakukan meggunakan serabut petola yang dirawat untuk perbandingan. Kepekatan akhir ion fosfat ditentukan berdasarkan keadah asid askorbik. Hasil kajian menunjukkan jerapan fosfat pada kedua-dua bahan penjerap adalah pantas dalam masa 10 mini pertamat. Keseimbangan jerapan, walau bagainamapun, dicapai selepas 60 minit. Jerapan fosfat mematuhi dengan baik model kinetik tertib kedua. Jerapan pada serabut petola semula jadi mematuhi dengan baik isoterma Freundlich, manakala untuk serabut petola yang dirawat, mematuhi isoterma Langmuir. Kapasiti jerapan maksimum untuk serabut petola semula jadi dan dirawat ialah masing-masing 87.7  $\mu g/g$  dan 2500  $\mu g/g$ . Secara keseluruhannya, prarawatan meningkatkan kecekapan serabut petola untuk menyingkirkan ion fosfat dari larutan akua.



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# LIST OF SYMBOLS

P	phosphorus	
Ne	neon	
qc	amounts of phosphate adsorbed (mg/g) at equilibrium	
q	amount of phosphate adsorbed (mg/g) at time t	
K <sub>ad</sub>	the rate constant (1/min)	
qt	the amount of phosphate adsorbed (mg/g) at time t	
k	rate constant	
С	concentration of solute after adsorption process	
q <sub>m</sub>	the maximum adsorption that can take place in grams of	
	adsorbate per gram of adsorbent	
a	isotherm constant	
q	the amount adsorbed at equilibrium (mg/g)	
С	equilibrium concentration in solution (mg/L)	
K	constant	
SGF	sponge gourd fibre	
$M_1$	initial concentration of phosphate solution ( $\mu$ g/ml)	
V <sub>1</sub>	initial volume of phosphate solution (ml)	
$M_2$	final concentration of phosphate solution ( $\mu$ g/ml)	
V <sub>2</sub>	final volume of phosphate solution (ml)	
٦	wavelength	
V	volume of phosphate solution (ml)	
C,	initial concentration of phosphate (µg/ml)	
C <sub>f</sub>	final concentration of phosphate (µg/ml)	
W	weight of adsorbent (sponge gourd fibre)	
NSGF	natural sponge gourd fiber	
PSGF	pre-treated sponge gourd fiber	



### **CHAPTER 1**

#### INTRODUCTION

## **1.1 General Information**

One of the common pollutants in wastewater is phosphorus. Typically, raw domestic wastewater has total phosphorus concentration of approximately 10 mg/L, the principal forms of phosphate being orthophosphate, pyrophosphate, and tripolyphosphate, together with smaller amounts of organic phosphate (Snoeyink *et al.*, 1980). Excessive discharge of phosphorus into surface water stimulates growth of phosphate-dependent organisms such as algae and duckweed, causing eutrophication. These blooms deplete dissolved oxygen, killing fish and other aquatic animals (Namasivayam and Sangeetha, 2004). Such wastewater therefore need to be treated to remove phosphate prior to discharge.

. Treatment technologies for removal of phosphate include coagulationprecipitation, biological methods (Karaca *et al.*, 2006), reverse osmosis (Liu *et al.*, 2006) and adsorption (Namasivayam and Sangeetha, 2004). Adsorption is comparatively more useful and economic tecnique for phosphate removal. However, adsorption is dependent on several factors such as temperature, pH, types of adsorbent, contact time andadsorbent dose (Cooney, 1999; Namasivayam and Sangeetha, 2004). Various materials investigated as phosphate adsorbents include raw and calcinated dolomite (Karaca *et al.*, 2006), iron oxide tailings (Zeng *et al.*, 2004), activated carbon (Namasivayam and Sangeetha, 2004) and aluminum oxide hydroxide (Tanada, 2003). Ideally, the adsorbents need to be efficient, easily available and low cost (Namasivayam and Sangeetha, 2004).

Sponge gourd fibre (*Luffa cylindrical*) is a low cost and easily available vegetable biomass. Its biodegradability can contribute to a healthier ecosystem and its low cost and reasonable performance fulfill economic interests of various industries (Tanobe *et al.*, 2005). However, its application as an adsorbent is relatively unknown.

## 1.2 Objectives of Study

The objectives of this study are:

- a) To determine and characterize phosphate removal efficiency from aqueous solution by sponge gourd fibre.
- b) To determine the effect of chemical treatment of sponge gourd fibre on phosphate removal efficiency.

#### 1.3 Scope of Study

In this study, the ability of sponge gourd fibre as adsorbent for phosphate ion from aqueous solution was investigated at different contact time and initial phosphate concentration. Comparison was made with natural and modified sponge gourd fibre. The residual phosphate concentration in solution was determined calorimetrically using UV-VIS spectrophotometer.



#### **CHAPTER 2**

#### LITERATURE REVIEW

#### 2.1 Phosphorus

### 2.1.1 General characteristics

Phosphorus (P) is a multivalent non-metal of the nitrogen group. It is the  $15^{th}$  member in Periodic Table with atomic number 15 and atomic mass 30.98 g/mol. In its compounds, phosphorus exhibits oxidation number between -3 to +5. The electronic configuration for phosphorus is [Ne]3s<sup>2</sup>3p<sup>3</sup> (Roques, 1996).

Phosphorus can be exists in three main allotropic forms, namely white, red and black phosphorus. Other forms of solid phosphorus are mainly derived from these main allotropes. White phosphorus glows in the dark, is spontaneously flammable when exposed to air and is a deadly poison. Red phosphorous can vary in colour from orange to purple due to slight variations in its chemical structure. The third form, black phosphorus, has the properties like graphite, that is it has the ability to conduct electricity. Naturally, phosphorus does not occur in free state. It is almost always found in its highest oxidation state (+5) in the form of orthophosphates.

#### 2.1.2 Phosphorus in water and wastewater

In nature, phosphorus is present in a variety forms. This includes organic phosphate, inorganic phosphate (orthophosphate) and polyphosphate (particulate P). Phosphate in water originates mainly from weathering of rocks while the phosphate in wastewater can be due to various anthropogenic sources, including sewage sludge and agricultural wastes. Approximately 50% to 70% of phosphorus in wastewater is orthophosphates (Roques, 1996), while the remainder being polyphosphate and organic phosphorus compound.

Orthophosphate consists mainly of H<sub>2</sub>PO<sub>4</sub><sup>-</sup>, HPO<sub>4</sub><sup>2-</sup> and PO<sub>4</sub><sup>3-</sup>. The distribution of each species is pH dependent (Figure 2.1). Typically, the dominant species are H<sub>2</sub>PO<sub>4</sub><sup>-</sup> and HPO<sub>4</sub><sup>2-</sup>, in particular at pH ~7.0. The dissociation of the various species are as follows: H<sub>3</sub>PO<sub>4</sub>  $\Longrightarrow$  H<sub>2</sub>PO<sub>4</sub><sup>-</sup> + H<sup>+</sup>; pK<sub>1</sub> ~ 2.16 H<sub>2</sub>PO<sub>4</sub><sup>-</sup>  $\Longrightarrow$  HPO<sub>4</sub><sup>2-</sup> + H<sup>+</sup>; pK<sub>1</sub> ~ 7.16 HPO<sub>4</sub><sup>2-</sup>  $\Longrightarrow$  PO<sub>4</sub><sup>3-</sup> + H<sup>+</sup>; pK<sub>1</sub> ~ 12.40



Figure 2.1 pH dependent variation of phosphorus speciation.

#### 2.1.3 Effect of excessive phosphate in water

Excessive of phosphate in water will cause eutrophication. Being a nutrient element, excessive phosphate stimulates the growth of plant and cause algal blooms. This will used up the oxygen in the water bodies.

The breakdown of dead algal biomass also involved the consumption of oxygen gas. Overall, this will cause the depletion of oxygen in the water system, which can adversely affect aquatic life and subsequently result in reduction of biodiversity (Oguz, 2004).

#### 2.2 Technologies for Phosphate Removal from Wastewater

Since phosphorus bring a serious impact to the environment, wastewaters that containing phosphorus need to be treated prior to discharge. Technologies for phosphate removal can be divided into three categories, namely physical, biological and chemical methods. Physical technologies include reverse osmosis, electrodialysis, anion exchange and adsorption (Namasivayam and Sangeetha, 2004). Biological process is based on the application of algae, bacteria or plants under anaerobic conditions (Wang *et al.*, 2007) while chemical removal technology include chemical precipitation method. The chemicals normally used in precipitation are alum, lime, and iron salts.



#### 2.2.1 Adsorption

Adsorption is a process by which ions or molecules present in solution phase condense or concentrate on the surface of a solid phase. The material being concentrated is known as the adsorbate while the adsorbing solid is known as adsorbent (Figure 2.2).



Figure 2.2 Interaction between adsorbate and adsorbent during adsorption.

There are two main types of adsorption, the physical adsorption and chemical adsorption. Physical adsorption involves weak attraction or Van de Waals forces between the adsorbate and adsorbent. The adsorbed molecule is not fixed to particular site, but free to move over the surface. Physical adsorption is quite reversible, that is with a decrease in concentration the adsorbed material can desorbed at the same extent to the amount adsorbed. Chemical adsorption involves chemical bonding between the adsorbate and adsorbent. Thus, chemical adsorption is said to be irreversible. Normally the adsorbed molecule forms a layer over surface which is one molecule thick and the capacity of the adsorbent is considered to be exhausted when its surface is covered by monomolecular layer.

### 2.2.2 Electrodialysis

Electrodialysis consists of applying direct current across a body of water which is separated by a membranes that is only permeable to cations and anions. In this process,

cations move toward cathode while anions move toward anode. Water containing phosphate alternates with those from which have been removed. The water in the substance-enriched layers is recirculated to a certain extent to prevent excessive accumulation of the substances. Experience with pilot plants indicates that electrodialysis can be removed up to 50% of the dissolved inorganics (Manahan, 2000).

#### 2.2.3 Reverse osmosis

Reverse osmosis functions by forcing pure water through a semi permeable membrane that allows only the passage of water but not other materials. When the wastewater is subjected into this process, the phosphate will remains on the membrane and thus it can be separated (Manahan, 2000).

### 2.2.4 Anion exchange

Anion exchange involves the reversible transfer of ions between aqueous solution and a solid material capable of bonding ions. In this process, the water is passed over an anion exchanger and the phosphate anion is exchange with the exchanger, yielding pure water. The anion can be removed when the anion exchanger is regenerated. The reaction can be represented as follows:

 $OH^{-+}(An(s)) + H^{+} + X^{--+} X^{-+}(An(s)) + H_2O$ where  $^{+}(An(s))$  represent the solid anion exchanger.



The advantage of this technique is it can produce water with high quality. However, microbial growth can diminish the exchanger's efficiency (Manahan, 2000).

#### 2.2.5 Precipitation

Precipitation is a chemical process where the precipitant(s) will react with phosphate ion to form a precipitate that is easily removed by filtration. The common precipitants and their products are shown in Table 2.1. Among the precipitants, lime,  $Ca(OH)_2$  is the most commonly used and the reaction involved is as follows:

$$5Ca(OH)_2 + 3HPO_4^{2-} \longrightarrow Ca_5OH(PO_4)_3 + 3H_2O + 6OH^{-1}$$

The hydroxyapatite,  $Ca_5OH(PO_4)_3$ , that formed can then be removed simply by filtration to yield a pure water. Precipitation processes can be removed at least 90-95% phosphorus at reasonable cost (Manahan, 2000).

Precipitant(s)	Products
Ca(OH) <sub>2</sub>	Ca <sub>5</sub> OH(PO <sub>4</sub> ) <sub>3</sub> (hydroxyapatite)
Ca(OH) <sub>2</sub> + NaF	$Ca_{5}F(PO_{4})_{3}$ (fluorapatite)
Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	AlPO4
FeCl <sub>3</sub>	FePO <sub>4</sub>
MgSO <sub>4</sub>	MgNH <sub>4</sub> PO <sub>4</sub>

Table 2.1 Chemical precipitants for phosphate and their products.

## 2.3 Removal of Phosphate by Adsorption

Among all the techniques for phosphorus removal, adsorption method is more preferred because of its low cost, ease of equipment use, lower sludge production and the potential of recycling retained phosphorus (Wang *et al.*, 2007). Due to these factors, adsorption had been widely investigated for treatment of industrial wastewater.

### 2.3.1 Adsorbent investigated for phosphorus removal

The adsorbents that have been investigated for phosphorus removal are shown in Table 2.2. The adsorption process among other is dependent on pH, temperature, time, concentration and adsorbent dosage (Cooney, 1999).

Adsorbent	References
Fly ash	Agyei et al.,2002
Alunite	Ozacar, 2003
Aluminium oxide hydroxide	Tanada et al., 2003
Iron oxide hydroxide	Zeng et al., 2004
ZnCl <sub>2</sub> activated coir pith carbon	Namasivayam and Sangeetha, 2004
Blast furnace slag	Oguz, 2004
Raw and calcinated dolomite	Karaca et al., 2006
Modified palygorskites	Ye et al., 2006
Lithium intercalated gibbsite	Wang et al., 2007
Natural and surface modified coir pith	Krishnan et al., 2008
Mesoporous ZrO <sub>2</sub>	Liu et al., 2008
Sludge from fuller's earth	Yong et al., 2007

Table 2.2 Adsorbents used in phosphorus removal.

## 2.3.2 Adsorption kinetics

Adsorption kinetics refers to the effect of contact time on the adsorption process. Typically, the rate of adsorption is initially rapid, then slowed down and subsequently remained constant at equilibrium (Krishnan *et al.*, 2007; Namasivayam and Sangeetha, 2004). For example, Krishnan *et al.* (2008) reported that phosphate removal by natural and surface modified coir pith was rapid during the first 3 hours. After that, the rate slowed down until equilibrium was attained after 12 hours (Figure 2.3). This trend is due to initially large number of surface site is available for adsorption process. The rate slowed down because the remaining site of the adsorbent is difficult to be occupied due to repulsion forces.



Figure 2.3 Effect of time on adsorptive phosphate removal by natural and surface modified coir pith.

The adsorption kinetics can be described by selected kinetic models, namely pseudo first order and pseudo second order models.

The pseudo first order model can be expressed as follows (Ozacar, 2003):

$$\log(q_e - q) = \log q_e - \frac{K_{ad}}{2.303}t$$

where  $q_e$  and q are the amounts of phosphate adsorbed (mg/g) at equilibrium and at time t (min), respectively, and  $k_{ad}$  is the rate constant (1/min). A linear graph of  $\log(q_e-q)$  vs t shows that the adsorption follows pseudo first order kinetics.

The pseudo second order model can be expressed as follow (Krishnan *et al.*, 2008):

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