



Asia  
around the  
world



## *Conference Proceeding*

**The analytica Vietnam Conference 2009**

## Preface

The two National Conferences of Vietnam Analytical Sciences Society (VASS) were successfully organized in 2000 and 2005 in Vietnam. This time Vietnamese scientists being concerned about the analytical science and technology have a chance to meet and exchange their recently latest research achievements at the International Conference on Analytical Sciences and Biotechnology called Analytica Vietnam Conference 2009. This Conference is an important event to mark the matured development of a multi-disciplinary field in Vietnam. Analytica Vietnam Conference 2009 will be considered as the third National Conference of VASS with the participation of many scientists coming from EU, China, Japan, Singapore and other regional countries.

This year we will organize a scientific conference in parallel with the First International Trade Fair for Analysis, Biotechnology, Diagnostics, Laboratory Technology and Services sponsored by Ministry of Science and Technology (MOST-Vietnam) and supported by International Fair and Exhibition Service Ltd. (IMAG) belonging to Munich Trade Fairs International Group (MMI) in Germany.

Together with the Trade Fair, the attendants will have a chance to join three separate workshops on (1) Technology Exchange and Related Support; (2) Understanding and Implementing ISO 17025 and (3) Analytica Vietnam Conference 2009.

It can be said that the Analytica Vietnam Conference 2009 is a significant event attracting about 200 participants from national and international research institutes, universities and scientific institutions. On behalf of the Organizing Committee, I would like to send the warmest greetings and best wishes to our distinguished guests to join the Conference this time and exchange their research results in the fields of analytical science and technology, biotechnology, food safety and clinical diagnostics.

Munich Trade Fairs International Group in general and International Fair and Exhibition Service Ltd. (IMAG) in particular is one of the World's Leading Trade Show Organizers. IMAG has organized 40 trade fairs on production materials, domestic and high technology products. Annually, over 30,000 exhibitors from more than 100 countries and two million visitors join the trade fairs in Munich. In addition, MMI have organized the trade fairs in Asia, Russia, Middle East and South America. MMI has its own global network with 4 subsidiaries and 64 representative offices in 86 overseas countries.

This time, MMI chose Vietnam as its destination in South East Asia. The Analytica Vietnam 2009 has marked the turning point and the recognition of international community to the potential market of Vietnam, especially when Vietnamese Government has offered the investment policies on scientific research, the establishment of key laboratories and the improvement in scientific equipment for research institutes nationwide.

As far as I know, it is the first time that Vietnam organizes a scientific conference in parallel with an equipment exhibition. The scientific conference is held together with the exhibition since the scientists are the main clients who will use the scientific equipment for their researches and then will create the advanced scientific works to meet the demand of the society.

I would like to take this opportunity to express my thankfulness to Research Centre of Environmental Technology and Sustainable Development (CETASD) belonging to Hanoi University of Science, Vietnam National University, Hanoi and Institute of Biotechnology (IBT) belonging to Vietnamese Academy of Science and Technology (VAST) who have made their great contributions to holding this Conference. I would also highly appreciate the coordination roles of Prof. Pham Hung Viet, Head of Department for International Affairs of VASS and currently the Co-Chairman of Organizing Committee, Prof. A. Ketzrap (Technical University of Munich) and Prof. Le Tran Binh (Former Director, IBT). In addition, we would highly appreciate the organizing committee members for their active works in managing the peer-reviewed activities of submitted papers.

I would like to say the thank you to all the scientists for contributing their research works to the Conference. The Conference Proceeding will be published including all the abstracts of submitted papers and selected English full-text papers with good peer-reviewed evaluation of Vietnamese and international

scientists. In coming time, the Organizing Committee will further consider and publish other full-text papers in a special issue of Vietnamese Journal of Analytical Sciences in accordance with the Journal requirements.

Once again, on behalf of the Organizing Committee and the International Advisory Board, I would like to send the warmest greeting and best wishes to all the participants and wish the conference success. Thank you very much.



Prof. Dr. Sc. Long Tu Minh  
President of VIASS  
Head of the International Advisory Board of Analytical Vietnam Conference 2009

15/10/2009, Hanoi, Vietnam

Dear friends,  
I am very pleased to hear that you are all well and hope you are enjoying the conference. I am sure you will have a very successful and enjoyable time in Hanoi. I am sure you will have a very successful and enjoyable time in Hanoi. I am sure you will have a very successful and enjoyable time in Hanoi.

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# Program for Analytica Vietnam Conference 2009

Hanoi, March 19-20, 2009

## March 19 morning: Analytical Session

8.00-8.30 Registration

8.30-8.40 Welcoming speeches

Session Ia: Chaired by Prof. Pham Hung Viet and Prof. Antonius Kettrup

8.40-9.10 Validation Analytical Methods and Procedures

*Dr. Ludwig Huber, Labcompliance Ltd, Oberkirch, Germany*

9.10-9.30 Some Experience in Trace Analysis of Organic Compounds by Chromatographic Techniques

*Prof. Dr. Chu Pham Ngoc Son, Centre for Education and Development of Chromatography (EDC), Hochiminh City, Vietnam*

9.30-10.00 New Application Possibilities of High Resolution Continuum Source AAS (HR-CS AAS)

*Dr. Andrej Glomb, Analytik Jena AG, Jena, Germany*

10.00-10.20 Brominated Flame retardants in Vietnamese environment: Contamination status in the context of the Asia-Pacific region

*Dr. Tu Binh Minh, CETASD, Hanoi University of Science, Vietnam*

10.20-10.35 Coffee break

Session Ib: Chaired by Prof. Chu Pham Ngoc Son and Prof. Jiang Guibin

10.35-10.55 How to spoil a river? The Trend of Water Quality in the Thi Vai River

*Prof. Dr. Le Quoc Hung, Institute of Chemistry, VAST, Vietnam*

10.55-11.25 Reagent Free Ion Chromatography (RFIC) and its Application in Environmental Ion Analysis

*Dr. Joachim Weiss, Dionex Corporation, International Operations, Am Wirtzgarten 10, D-65510 Idstein, Germany*

11.25-11.45 Development of a Solid-Contact Ion-Selective Electrode for the Detection of Cadmium at Nanomolar Level

*Dr. Do Phuc Quam, CETASD, Hanoi University of Science, Vietnam*

11.45-12.05 Development of Mercury-free Metal Film Electrode for Stripping Voltammetric Measurement of Trace Heavy Metals

*Assoc. Prof. Nguyen Van Hop, Department of Chemistry, Hue College of Sciences, Hue University, 77 Nguyen Huu Str., Hue City, Vietnam*

12.05-13.00 Lunch

## March 19 afternoon: Analytical Session (Cont.)

Session Ic: Chaired by Assoc. Prof. Nguyen Van Hop and Dr. Joachim Weiss

13.00-13.30 Impurities in Herbs

*Dr. Geroalf Titze, Institute for Pharmaceutical Analysis and Contract Research, Am Haag 4, D 82166 Grafelfing/Munich, Germany*

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## FLUORIDE REMOVAL PERFORMANCE OF LATERITE ADSORBENT

Hoang Trong Si

College of Medicine and Pharmacy, Hue University

Dang Thi Thanh Loc, Dinh Quang Khieu

College of Sciences, Hue University

### ABSTRACT

*Laterite is used as an adsorbent for removal of fluoride from aqueous solution. The laterite was characterized by XRD, SEM and isotherm adsorption of nitrogen. Batch kinetics and isotherm studies were carried out as a function of contact time, fluoride concentration and pH. The necessary time to reach equilibrium was 40 minutes as the initial concentration of fluoride is in the range of 5 to 15 mg/L. The fluoride removal was the highest at pH 7. The Freundlich and Langmuir adsorption models were used for the mathematical description of adsorption equilibrium and it was found that the experimental data fitted well to the Freundlich model. Both adsorption models, based on the assumption of the pseudo-first-order and pseudo-second-order models, were also applied to examine the kinetics of the adsorption. Results indicated that the process followed pseudo second-order kinetics.*

*Key words: laterite, adsorption equilibrium, Freundlich model, pseudo second order equation*

### 1. INTRODUCTION

It is found that long-term ingestion of fluoride rich drinking water leads to serious health disorder, the fluorosis. Release of fluoride in ground water occurs due to both natural dissolution of minerals from subterranean strata and industrial discharge. Fluorosis results in molting of teeth, softening of bones, ossification of tendons and ligaments and also the neurological damage in severe situation [1]. World Health Organization has set a guidance value of  $1.5 \text{ mg L}^{-1}$  for fluoride in drinking water [2]

Adsorption has been considered as the most efficient and applicable technology for fluoride removal from drinking water compared to the technologies like reverse osmosis, nanofiltration, electrodialysis. Activated alumina, activated carbon and some other cost materials have been used as fluoride adsorbents [3]. Among these adsorbents, some inorganic mineral containing iron oxide such as diatomite, clay, and laterite have been intensively studied. In present paper, the laterite, the red soil, available in plenty in different parts of Vietnam was studied on the removal of fluoride from aqueous solution,

### 2. EXPERIMENTAL

Laterite from Quang tri province-Vietnam was used as an adsorbent. Stock solution of sodium fluoride ( $\text{NaF}$ , Merck) of desired concentration was prepared by dissolving appropriate quantity of sodium fluoride in distilled water.

A textural property of laterite was determined by nitrogen adsorption/desorption isotherms using Micromeritics at 77K. The phase of laterite was identified by X-ray diffraction (XRD), recorded on 8D Advance Buckler, Germany with  $\text{CuK}\alpha$  radiation with a scan step size of  $0.01^\circ$  and a scan step time of 0.04s. Morphology of laterite was observed by Scanning Electron



Microscope (SEM, JSM-5300 LV). Element analysis was conducted by Electron Dispersed Spectrometry (EDS/SEM, JMS-5300 LV).

In the both adsorption process fluoride solution of known concentration was agitated with laterite in an incubator. The experiments were conducted in thermostat in  $25^{\circ}\text{C}\pm 1$ . The solution was filtered and the fluoride concentration in the filtrate was measured by standard method (SPAND – Reagent). The adsorption capacity ( $q_t$ ) was calculated using the Eqn. (1)

$$q_t = \frac{(C_0 - C_t)}{m} \times V \quad (\text{mg/g}) \quad (1)$$

where  $C_0$  and  $C_t$  are fluoride concentration (mg/L) at initial time and the time recorded;  $V$  the volume of solution (L) and  $m$  weight of the adsorbent (g).

Kinetic experiment was done in three initial concentrations of fluoride. Initial fluoride concentrations were 5, 10, 15 mg/L. The laterite is 1g/50 mL for each experimental. Initial pH of solution was 7. The experimental was continued for 180 minutes and samples were drawn from the mixture at predetermined time intervals for analysis. Kinetic of fluoride sorption was modeled by the pseudo first-order, pseudo second-order equations presented below as Eqn. (2)-(3), respectively:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (2)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (3)$$

where  $q_e$  and  $q_t$  are the adsorption capacities (mg/g) at equilibrium at time recorded, respectively;  $C_e$  is the fluoride concentration at equilibrium;  $k_1$  is the pseudo first-order rate constant of adsorption ( $\text{min}^{-1}$ );  $k_2$  the pseudo second-order rate constant of adsorption ( $\text{g}^2/\text{mg}^2 \cdot \text{min}^{-1}$ ) [4].

Table 1. Adsorption data for isotherm at  $C_0=10 \text{ mg/l}$  and different  $m/V$  ratio

$m$ (g/L)	$C_e$ (mg/L)	$q$ (mg/g)	$1/C_e$	$1/q_e$	$\log C_e$	$\log q_e$
3	7.42	0.86	0.13	1.16	0.87	-0.07
4	6.64	0.84	0.15	1.19	0.82	-0.08
5	6.12	0.78	0.16	1.29	0.79	-0.11
6	5.40	0.77	0.19	1.30	0.73	-0.12
10	2.79	0.72	0.36	1.39	0.45	-0.14
15	1.36	0.58	0.74	1.74	0.13	-0.24
20	0.57	0.47	1.74	2.12	-0.24	-0.33

Kinetic experiments presented that maximum time required to reach equilibrium was 40 minutes; therefore, the equilibrium time for equilibrium experiments was chosen 40 minutes. The initial fluoride concentrations and the corresponding amount of laterite in order to study isotherm adsorption was listed in table 1. The isotherm of fluoride adsorption was analyzed using the Freundlich and Langmuir [5].

The Freundlich equation is linearized in the form:

$$\ln q_e = \ln K_f + \frac{1}{n} C_e \quad (4)$$

where  $C_e$  is equilibrium concentration of fluoride (mg/L);  $K_f$  and  $n$  are indicators of sorption capacity and sorption intensity, respectively.

The Langmuir equation is linearized in the form:

$$\frac{1}{q_e} = \frac{1}{bq_m C_e} + \frac{1}{q_m} \quad (5)$$

where  $q_m$  is the maximum capacity of adsorption (mg/g) and  $b$  is a constant related to the affinity of the binding sites (mL/g).

### 3. RESULTS AND DISCUSSION

#### 3.1. Characterization of laterite

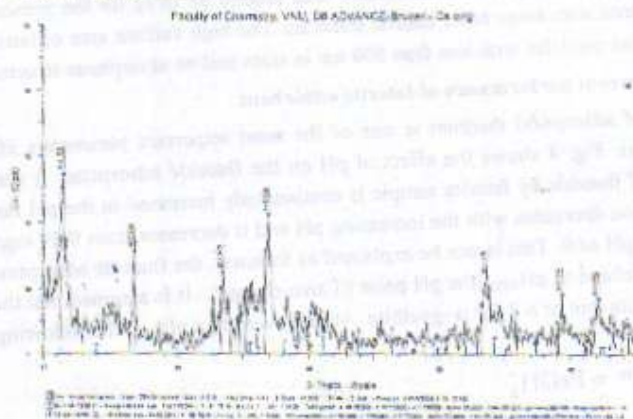


Fig. 1. XRD of laterite.

Fig. 1 shows XRD of laterite. It shows that the present laterite poses amorphous-structure. Besides, the small amount of crystalline phase could be identified as goethite  $\alpha$ -FeO(OH) (pattern: 01-081-0464) and of akaganeite-FeO(OH) (pattern: 00-034-1266).

The elemental composition of laterite was analyzed by EDS. As can be seen from table 2, the main composition of studied laterite is iron oxides. The laterite with large amount of iron oxides should be expected the high capacity of adsorption for metal ions and other anions.

Table 2. The elemental compositions of laterite

SiO <sub>2</sub> (%)	Al <sub>2</sub> O <sub>3</sub> (%)	Fe <sub>2</sub> O <sub>3</sub> (%)	CaO (%)	MgO (%)	Ignition loss (%)
20.3	19.5	36.2	1.40	3.40	12.00



Fig. 2. SEM observation of laterite

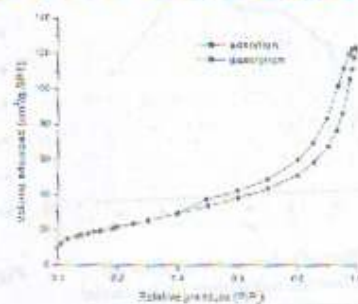


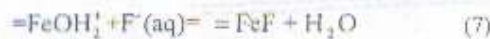
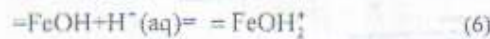
Fig. 3. Nitrogen isotherm for laterite



Fig.2 presents SEM observation of laterite. As can be seen from Fig.2, the laterite consists of very fine particles less than 500 nm. The textural properties of laterite were also studied by nitrogen adsorption isotherm. Fig. 3, gives the adsorption and desorption isotherms of N<sub>2</sub> at 77K on laterite sample. The obtained curve is the isotherms of III type, its shapes is typical of macroporous materials. This sample exhibits one well-expressed hysteresis loop at P/P<sub>0</sub> >0.4 corresponds to capillary condensation in the inter-particle pores. The nitrogen physisorption at low relative pressure (P/P<sub>0</sub> <0.3) is usually assigned to monolayer adsorption. Therefore the data collected in this part of the isotherm are used for the calculation of surface area of material with BET method [6]. Thus calculated surface area around 88 m<sup>2</sup>/g for the present laterite is very high compared with some other natural minerals. The high surface area of laterite has also agreed with its fine particles with less than 500 nm in sizes and its amorphous structure.

**3.2. Fluoride removal performance of laterite adsorbent**

The initial pH of adsorption medium is one of the most important parameters affecting the adsorption process. Fig. 4 shows the effect of pH on the fluoride adsorption. It was observed that the uptake of fluoride by laterite sample is continuously increased in the pH range of 2-7. Then the adsorption decreases with the increasing pH and it decreases from 0.49 mg/g at pH of 7 to 0.42 mg/g at pH of 9. This can be explained as follows: the fluoride adsorption has been considered to be related to pH<sub>ZPC</sub> (the pH point of zero charge). It is assumed that the pH<sub>ZPC</sub> of laterite matches with that of 6.8 for α-goethite. At pH lower than pH<sub>ZPC</sub> the following reactions are suggested [7].



As a result, the uptake of fluoride increases with the increase in pH due to the electro-static force. At pH > pH<sub>ZPC</sub>, the surface of laterite passes negative charge and tends to repel the fluoride ions via columbic repulsion. In addition, the competition between hydroxyl ions and fluoride the active adsorption sites also contribute to the decrease of fluoride uptake under alkaline conditions.

The adsorption data for the uptake of fluoride versus contact time at different initial concentrations are presented in Fig. 5. Kinetic parameters of kinetic modeling of fluoride adsorption are shown in Table 2.

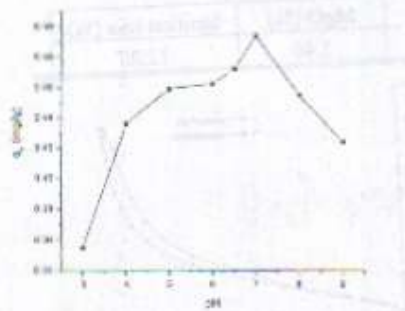


Fig. 4. Effect of solution pH on fluoride adsorption (fluoride adsorption under initial fluoride concentration of 10 mg/l, adsorbent dose, 1g/50 mL, equilibrium time, 40 min and

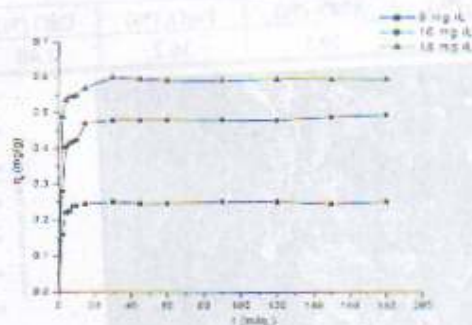


Fig 5. Effect of initial fluoride concentration on the adsorption kinetics of fluoride by laterite



temperature, 25°C)

Table 2. Kinetic parameters for the adsorption of fluoride onto laterite

The pseudo-first order equation (Lagergren equation)				
$C_0$ (mgF/L)	$R^2$	$K_1$ (min <sup>-1</sup> )	experimental $q_e$ (mg/g)	theoretical $q_e$ (mg/g)
5	0,89	0,21	0,25	0,09
10	0,89	0,20	0,48	0,24
15	0,89	0,09	0,60	0,11
The pseudo-second order equation				
$C_0$ (mgF/L)	$R^2$	$k_2$ (mgg <sup>-1</sup> min <sup>-1</sup> )	experimental $q_e$ (mg/g)	theoretical $q_e$ (mg/g)
5	1	4,63	0,25	0,26
10	1	1,49	0,48	0,50
15	1	2,10	0,60	0,61

According Fig. 5, fluoride uptake is relatively fast for all the concentration studied. At initial fluoride concentration in range from 5-15 mg/L, the system reached to equilibrium within 40 min. The pseudo second-order described the adsorption with high correlation coefficient ( $R^2 = 1$ ) and better than the pseudo-first order equation. The rate constants of the pseudo second-order rate equation for fluoride adsorption were obtained 4.63, 1.49 and 2.1 mg/g.min at initial fluoride concentrations of 5, 10 and 15 mg/L, respectively. The theoretical  $q_e$  values were closer to the experimental  $q_e$  values. In the view of these results, it can be said that the pseudo-second-order kinetic model provided a good correlation for the adsorption of fluoride onto laterite in contrast to the first-order-model.

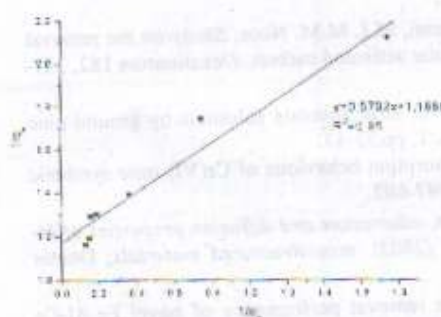


Fig. 6. Langmuir plot for the adsorption of fluoride onto laterite

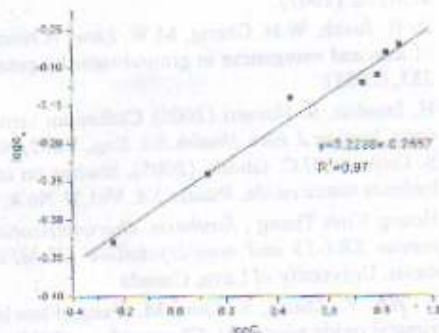


Fig. 7. Freundlich plot for the adsorption of fluoride onto laterite

Fig. 6-7 show isotherm modeling of fluoride adsorption by linear plots of Freundlich and Langmuir. The maximum adsorption capacities ( $q_m$ ) and other constants were shown in Table 3. Isotherm data are basic requirements for the design of adsorption reactors, moreover analysis of adsorption isotherm is important to develop an equation which accurately represents the results and which can be used for design purposes. The Freundlich model described the isotherm of

fluorite adsorption with high correlation coefficient ( $R^2=0.97$ ) and better than Langmuir model. In fact, Langmuir equation is based on the assumption that maximum adsorption corresponds to a saturated monolayer of solute on the adsorbent surface, that energy of adsorption is constant and that there is no transmigration of adsorption in the plane of the surface while the empirical Freundlich model based on sorption on a heterogeneous surface. Hence, this is compatible with the fact that laterite possesses "rough" surfaces and its walls contain many micropores.

Table 3. Isotherm parameters of fluorite adsorption by laterite

Langmuir model (Linearized shape)			Freundlich model (Linearized shape)		
$q_m$	$b$	$R^2$	N	$K_f$	$R^2$
0.856	2.018	0.95	4.488	0.539	0.97

#### 4. CONCLUSIONS

In this study, the adsorption of fluorite from aqueous solution was investigated using laterite as an adsorbent. The results indicated that adsorption capacity of the adsorbent was considerably affected by initial pH, initial fluoride concentration, contact time. The results showed that the amount of fluorite adsorbed increased with increasing initial fluoride concentration and uptake of fluoride took place at a pH in the range 2-7. Then, the adsorption of fluoride decreased with increasing pH. Equilibrium data fitted well in a Freundlich isotherm equation. The pseudo-second-order kinetic model provided the better correlation for the adsorption data.

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