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Simple On-Line Preconcentration Spectrophotometry for Detecting Lead Contamination from Drinking Water Coolers and Glazed Bowl Samples

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Lead is known as a toxic metal and can cause extensive damage for human health. Hence, the determination of lead is necessary and this could help raise public awareness of lead toxicity. A simple on-line spectrophotometric system for the determination of lead at a trace level with on-line preconcentration step using Amberlite IR-120 as a sorbent packed in a minicolumn was developed. It is based on Pb(II)-4-(2-Pyridylazo)-resorcinol (PAR) complex formation flowing along with ammonia buffer pH 10 to be detected spectrophotometrically at 519 nm. Under the optimal conditions, the linear range of 70-1000 $\mu\text{g l}^{-1}$ with a correlation coefficient (R^2) of 0.9998 was obtained. The limit of detection (LOD, based on $3S_{x/y}/m$) of 55 $\mu\text{g l}^{-1}$ and the relative standard deviation (RSD) within 5% (at 70 and 500 $\mu\text{g l}^{-1}$, $n = 10$) were achieved with an enrichment factor of 47 at 4 min loading time. The column can be reused up to 84 cycles. The proposed method was successfully applied for detecting Pb(II) contamination in drinking water samples discharged from water coolers and leachates from glazed bowls with satisfactory recoveries within the range of 90-107%.

Keywords: Lead, Spectrophotometry, On-line preconcentration, Glaze, Drinking water cooler

INTRODUCTION

Lead has been widely used in various industries for a number of products such as batteries, pigments, ceramics, petrol compounds, varnishes and lubricants [1-3]. It is also frequently used as a material for plumbing systems, for instance in drinking water cooler. If the water is in contact with plumbing systems for a long time, lead can be leached to the water. Another typical material containing lead is glaze which is a thin layer of liquid put on a piece of bowl and becomes hard and shiny when the bowl is heated in a hot oven. Therefore, there is a possibility of lead elution from the surface in some of glazed ceramic (dinnerware) when it is in hot or acidic condition [4]. Lead can cause a wide range of adverse effects on human health, particularly cardiovascular and reproductive system, kidney and lung [2]. Hence, the monitoring of lead concentration in such samples is of

importance in addition to an increasing recognition to assure its level does not exceed the value causing harm to human health.

There are various methods employed for lead determination in a variety of samples. Differential pulse anodic stripping voltammetry (DPASV) is a well-known analytical technique which is very suitable for quantitation of trace metals owing to its remarkable analytical sensitivity, relative instrumental simplicity, low equipment and maintenance cost as well as minimum sample pretreatment prior to analysis [5-7]. Another alternative for lead determination is spectrometry including inductively coupled plasma atomic emission spectrometry (ICP-AES) [8], inductively coupled plasma-mass spectrometry (ICP-MS) [9], flame atomic absorption spectrometry (FAAS) [10-11], and electrothermal atomic absorption spectrometry (ETAAS) [12-13] with the disadvantages of high purchase and maintenance cost, complicated operation and the requirement of well-controlled experimental conditions. UV-visible spectrophotometry is also an option that has been widely used as a detection technique

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for lead determination due to low cost, simplicity, speed, precision and accuracy. The technique is normally based on the formation of colored-complexing compounds with chromogenic reagents followed by measuring the absorbance at specific wavelength [14-15] but not at trace level. Therefore, preconcentration step is required to overcome the drawback. To serve this purpose, a number of techniques normally applied include solvent extraction [16], coprecipitation [17-18] and solid phase extraction [19]. Solid phase extraction is more interesting than others due to the fact that it uses less solvent volume, less contamination in the environment, greater times of reuse and higher preconcentration factor. In addition, various sorbents such as Amberlite XAD-2000 [19], Pb-Spec resin [20], Amberlite XAD-1180 [21], and Amberlite IR-120 [22] were employed for the preconcentration of lead. Owing to the advantages regarding thermal, mechanical and chemical stability under various experimental conditions, Amberlite IR-120 (cation exchange resin) is a suitable sorbent to be packed in a minicolumn. Furthermore, on-line system is also applied to help improve analytical performance due to its ease of use and low reagent consumption [23-24]. Here, the cost effective, simple and rapid method for the determination of lead at a trace level with on-line preconcentration step using a sorbent was developed based on Pb(II)-4(2-pyridylazo) resorcinol complex formation [14-15,25] and the detection by spectrophotometric detector at 519 nm.

EXPERIMENTAL

On-line Preconcentration System and Operation Procedure

The on-line preconcentration system was set up as shown schematically in Fig. 1. It consisted of two peristaltic pumps, P1 and P2 (Ismatec, Switzerland), a 6-port injection valve (Upchurch, USA), a spectrophotometer (Milton Roy Company, Italy) as a detector with a flow through cell and a chart recorder (Pantos Nippon Denshi Kagaku, Japan) as a readout unit for flow injection signal in the form of a peak. The sorbent was packed in a minicolumn (i.d., 0.4×1.0 cm) from Perspex. Each end of the minicolumn was fitted with cotton to support the sorbent material. Mixing coils were made of PTFE tubing. Certain parameters including the type

of sorbent, the length of minicolumn, pH for adsorption and the type of eluent were optimized using Flame Atomic Absorption Spectrometer (FAAS) 1600A (Perkin Elmer, USA).

At the load position of the 6-port injection valve, the minicolumn was first pre-conditioned by flowing ammonia-ammonium chloride buffer solution by P1 for 2 min. Afterwards, 3-way valve was switched and the sample/standard was drawn through the minicolumn packed with the sorbent on which Pb(II) ion was adsorbed. Then, the 6-port injection valve was switched to the injection position. Resulting adsorbed Pb(II) was eluted and mixed with the complexing agent and ammonia-ammonium chloride buffer solution with the continuous flow by P2. Finally, the complex of Pb(II) formed was monitored at 519 nm and the peak height was measured. The 6-port injection valve was switched back to the load position again to start new cycle of lead determination.

Chemicals and Reagents

All reagents were of the analytical reagent grade and used without further purifications. De-ionized water was used for reagent preparation. All plasticware and glassware were immersed in 10% (v/v) HNO₃ for 24 h followed by thoroughly rinsing with de-ionized water. Each stock standard solution was kept in a polyethylene bottle and stored in a refrigerator (4 °C) for a month. Pb(II) solution was prepared by diluting 1,000 mg l⁻¹ Pb(II) stock solution (Merck, Germany) with de-ionized water. The stock of 1.5×10^{-2} M PAR was prepared by dissolving 0.1619 g of 4-(2-Pyridylazo) resorcinol with ethanol and the volume was made up to 50 ml with ethanol. Buffer solution pH 10 was prepared by dissolving 2.4050 g of ammonium chloride with 33.70 ml conc. NH₄OH and the solution was adjusted to pH 10 with HCl and finally diluted to 250 ml with de-ionized water.

Sample Preparations

Drinking water samples were collected from public drinking water coolers in a dormitory and a cafeteria at Prince of Songkla University, two different sites from each area. The average pH of water samples is 6.8. Three types of glazed bowls were purchased from a local market in Hatyai, Songkhla. To store the drinking water sample, a polyethylene bottle was washed and soaked in 10% (v/v)

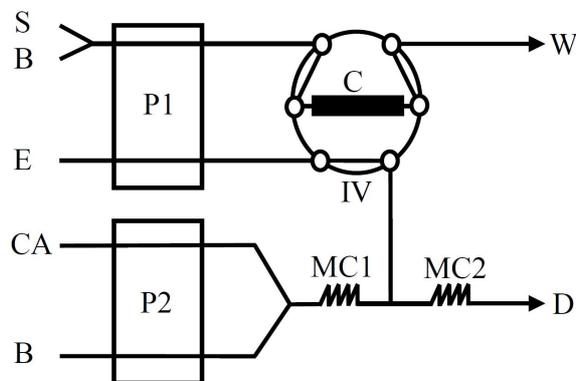


Fig. 1. The set up of the on-line preconcentration system for Pb(II) determination. Standard/sample (S), eluent (E), complexing agent (CA), ammonia-ammonium chloride buffer solution (B), peristaltic pumps (P1 and P2), 6-port injection valve (IV), minicolumn (C), mixing coils (MC1 and MC2), waste (W), detector (D).

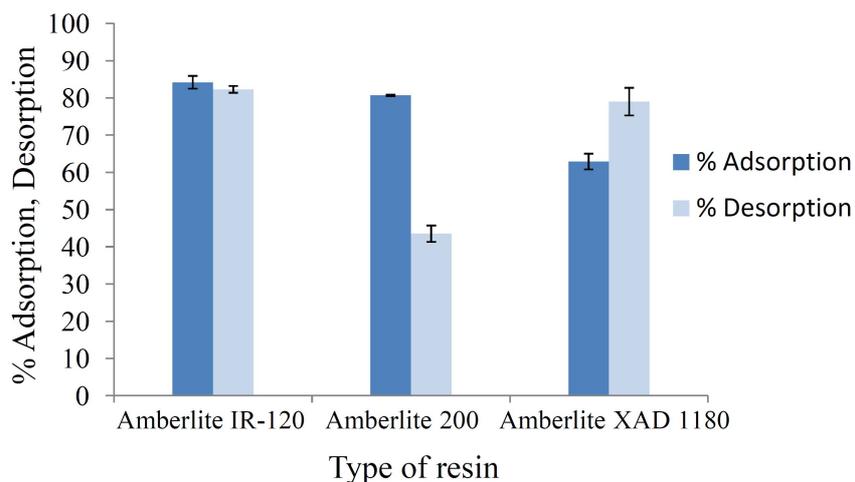


Fig. 2. Effect of sorbent type on 10 mg l⁻¹ Pb(II) determination (1.0 cm minicolumn, adsorption pH 10, 0.6 M, HCl as eluent, 1.30 ml min⁻¹ eluent flow rate, buffer pH 10, 0.2 mM PAR, 0.30 ml min⁻¹ PAR and buffer solution flow rate, 40 cm MC1, 80 cm MC2 and 4 min loading time).

HNO₃ for at least 24 h and then rinsed with de-ionized water. The volume of approximately 2 l of the sample was collected from each area. In order to preserve the sample, it was acidified by adding 2 ml of conc. HNO₃ into 2 l water sample (pH ≤ 2). All sample bottles were kept in a refrigerator at 4 °C. To extract Pb(II), three types of glazed bowls were filled with 4% acetic acid solution and allowed to stand in the dark for 24 h.

RESULTS AND DISCUSSION

Investigation of the Absorption of Pb(II)-PAR Complex

The absorption spectrum for the Pb(II)-PAR complex was recorded from 300 to 800 nm. The maximum absorption of the complex was observed at the wavelength of 519 nm, indicating the ability of the complex to absorb the light in

the visible range and be applied in spectrophotometric determination of Pb(II).

Sorbent and Minicolumn

Normally, the type of sorbent packed in a minicolumn is one of the parameters affecting sensitivity. Three different types, namely Amberlite IR-120, Amberlite-200 and Amberlite XAD-1180 were investigated with 10 mg l^{-1} Pb(II). Amberlite IR-120 and Amberlite-200 were found to have no significant difference on the percentage of adsorption with the values higher than those of Amberlite XAD-1180. However, the percentage of desorption of Amberlite-200 was very low compared with that of Amberlite IR-120 (Fig. 2). Therefore, Amberlite IR-120 was selected for further studies.

The length of minicolumn affecting the percentage of adsorption of 10 mg l^{-1} Pb(II) on the resin was varied in the range of 0.5-2.0 cm with 0.5 cm increment (figure not shown). The length of 1.0 cm gave the highest percentage and longer minicolumn caused the percentage to decrease dramatically due to back pressure and leakage in the system. Consequently, 1.0 cm-minicolumn was used for further studies.

pH for Adsorption

It has been reported that adsorption efficiency of Pb(II) on Amberlite IR-120 resin increased with increasing pH values due to less competitive adsorption between H^+ proton and Pb(II) on the resin surface in the batch system [26]. In this work, the adsorption pH for 10 mg l^{-1} Pb(II) on the resin was varied in the range of 7-10 and the best adsorption percentage was found at pH 10 (Fig. 3) which was chosen as an optimal condition.

Type and Concentration of Eluent

A number of eluents, including strong acids, have been used to strip adsorbed Pb(II) from different resins [27-30]. To follow the commonly used eluents, HCl and HNO_3 were selected for the investigation. It was observed that HCl gave the higher desorption percentage of Pb(II) than HNO_3 and it was employed as an eluent for this work. The concentration of HCl was varied in the range of 0.2-0.8 M. It was remarkable that the signal of Pb(II) apparently increased with increasing HCl concentration up to 0.6 M and then

reached the plateau (figure not shown). The concentration of 0.6 M was therefore selected to be suitable for this system.

Flow Rate of Sample and Eluent

In on-line systems, the flow rate manipulated by the peristaltic pump remarkably affects both adsorption and desorption percentages of Pb(II). The flow rate of sample/standard and eluent aspirated by P1 on the signal of 5 mg l^{-1} Pb(II) determination was varied from 1.00 to 1.50 ml min^{-1} , while that of P2 was kept constant at 0.30 ml min^{-1} . It was found that the higher the flow rate, the greater the signal (Fig. 4). The flow rate of 1.30 ml min^{-1} with the maximum signal was chosen for the subsequent experiments.

pH of Buffer Solution

The signal of Pb(II) determination also depends on the conditions of Pb(II)-PAR complex. Consequently, pH of buffer solution (B at P2) was optimized in the range of 8-12 due to the fact that, from the previous study [25], the complex is stable in basic conditions, especially at pH 10. The pH range of 9-12 was found to have no significant difference on the signal. However, the signal decreased dramatically at pH lower than 9 due to pH influence on the extent of complex formation (figure not shown). Therefore, pH 10 was selected.

PAR Concentration

The concentrations of PAR in the range of 0.07-0.30 mM were investigated. The peak height increased up to PAR concentration of 0.20 mM (figure not shown). The higher concentration of PAR led to complex formation at greater extent. However, slight decrease in peak height was observed at the concentration greater than 0.20 mM. This is probably due to the fact that excess amount of PAR could not react or mix well with the flowing stripped Pb(II). Therefore, 0.20 mM PAR was selected for further experiments.

Flow Rate of PAR and Buffer Solution

The flow rate of PAR and buffer solution which aspirated to the system by peristaltic pump (P2) was varied from 0.3 to 1.2 ml min^{-1} . The resulting peak height (Fig. 5)

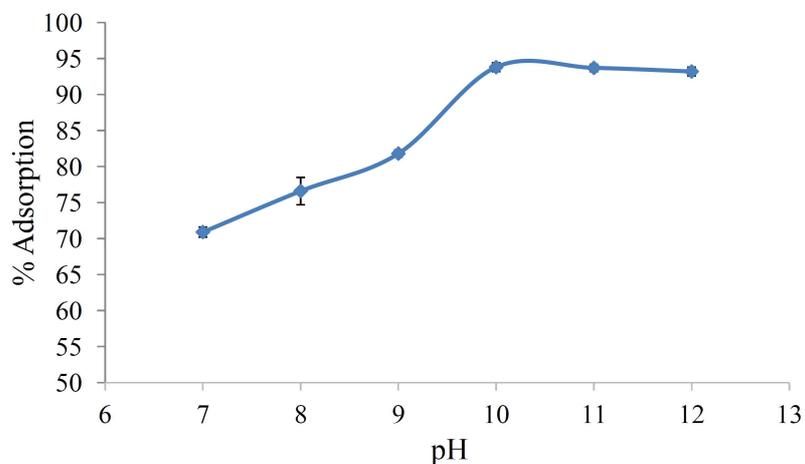


Fig. 3. Effect of pH on adsorption of 10 mg l^{-1} Pb(II) determination (1.0 cm minicolumn, 0.6 M HCl as eluent, 1.30 ml min^{-1} eluent flow rate, buffer pH 10, 0.2 mM PAR, 0.30 ml min^{-1} PAR and buffer solution flow rate, 40 cm MC1, 80 cm MC2 and 4 min loading time).

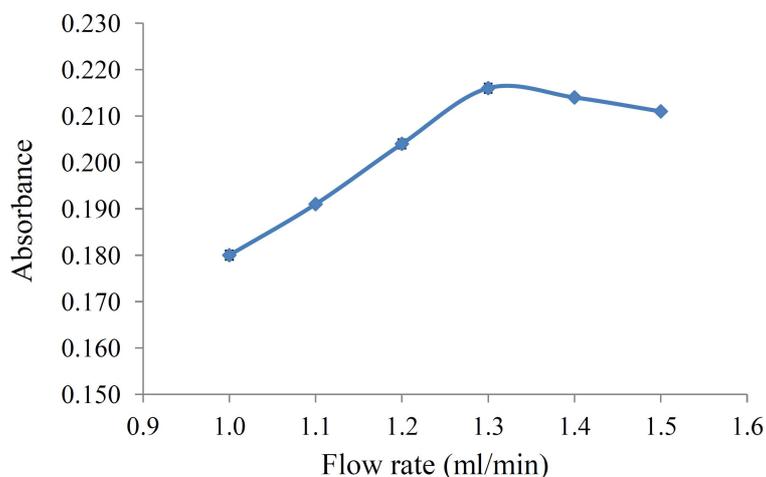


Fig. 4. Effect of Flow rate of sample/standard and eluent on 5 mg l^{-1} Pb(II) determination (1.0 cm minicolumn, adsorption pH 10, 0.6 M HCl as eluent, buffer pH 10, 0.2 mM PAR, 0.30 ml min^{-1} PAR and buffer solution flow rate, 40 cm MC1, 80 cm MC2 and 4 min loading time).

revealed the fluctuation of the signal intensity within a narrow range of flow rate due to the counterbalance effect of the mixing time and dispersion degree. Therefore, the lowest available flow rate of 0.30 ml min^{-1} was chosen for the subsequent experiments.

Length of Mixing Coils

Mixing coils, MC1 and MC2, were inserted into the

system for promoting the mixing of Pb(II) and the reagents. The PAR and buffer solution were first mixed at MC1 and then with eluted Pb(II) at MC2. In order to obtain the highest sensitivity, the length of MC1 and MC2 were optimized in the range of 20-100 cm (figure not shown). The peak height was found to increase up to 40 cm MC1 and then decrease at greater lengths owing to dilution, and therefore, 40 cm was chosen. The length of MC2 shorter

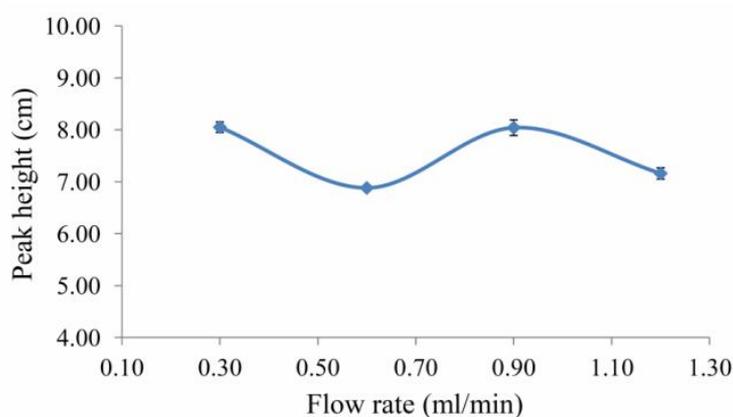


Fig. 5. Effect of flow rate of PAR and buffer solution on 1 mg l⁻¹ Pb(II) determination (1.0 cm minicolumn, adsorption pH 10, 0.6 M HCl as eluent, 1.30 ml min⁻¹ eluent flow rate, buffer pH 10, 0.2 mM PAR, 40 cm MC1, 80 cm MC2 and 4 min loading time).

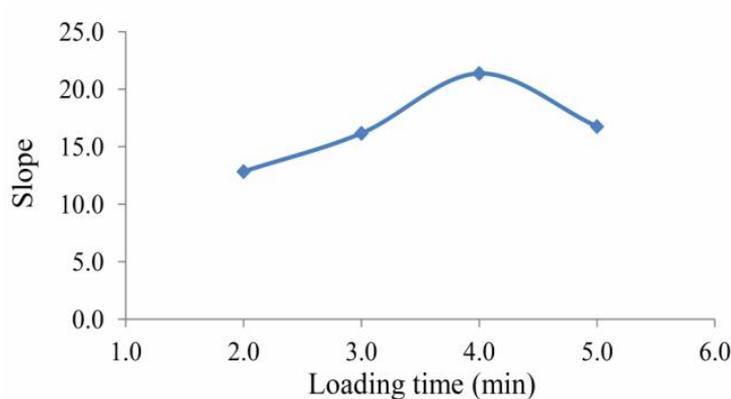


Fig. 6. Effect of sample loading time on Pb(II) determination (1.0 cm minicolumn, adsorption pH 10, 0.6 M HCl as eluent, 1.30 ml min⁻¹ eluent flow rate, buffer pH 10, 0.2 mM PAR, 0.30 ml min⁻¹ PAR and buffer solution flow rate, 40 cm MC1, 80 cm MC2).

than 80 cm caused lower peak height due to the fact that the complex between Pb(II) and PAR is not completely formed. The complex solution is more diluted at greater length, also resulting in lower signal. The MC2 length of 80 cm was thus chosen.

Sample Loading Time

The effect of loading time of sample was investigated by considering the slopes of calibration curve at different loading times with the results shown in Fig. 6. The

sensitivity increased with increasing loading time up to 4 min and then fell off at a longer time due to maximum sorption capacity of the resin. In this study, loading time of 4 min was selected in order to achieve the highest sensitivity.

Analytical Performance

The aforementioned optimal conditions for Pb(II) determination were employed to reveal the linear dynamic range of 70-1000 µg l⁻¹ with a good correlation coefficient

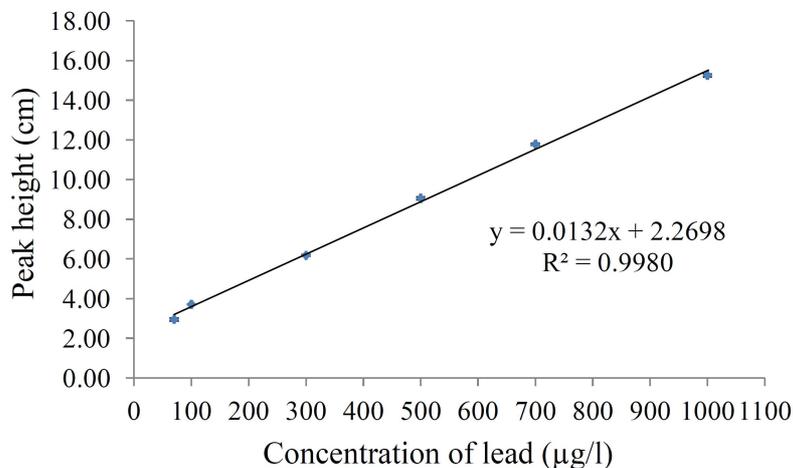


Fig. 7. The linear dynamic range of Pb(II) determination.

(R^2) of 0.9980 (Fig. 7). The LOD (based on $3S_{x/y}/m$) of $55 \mu\text{g l}^{-1}$ was obtained. Precision was presented in terms of percent relative standard deviation (%RSD) of 10 measurements which was found to be within 5% for both 70 and $500 \mu\text{g l}^{-1}$ Pb(II). The %recoveries, obtained by spiking Pb(II) at the concentrations of 300, 500 and $700 \mu\text{g l}^{-1}$ Pb(II) in drinking water and glazed bowl samples, were in the range of 90-107% (Tables 1 and 2), confirming that this method can be exploited for Pb(II) determination in real samples. The enrichment factor was investigated by the ratio of the slopes of calibration graphs with and without preconcentration which was found to be 47. The maximum 84 cycles of minicolumn of which the signal change not exceeding $\pm 5\%$ and the sample throughput of 8 samples/hour were achieved.

Interference Study

Potential interferences from ions commonly found in Pb(II) determination, especially Zn(II), Mn(II), Cu(II), Cr(III), Co(II), Ni(II), Cd(II), As(III), Fe(II), Al(III) and Hg(II) were investigated by adding interfering ions at various concentrations to standard solution of Pb(II). The 5% increase of peak signal was used as a criteria that the ion interferes the system. Possible potential interferences for the proposed method were found to be Zn(II), Mn(II), Cu(II) and Cr(III) with the interfering levels between $200\text{--}300 \mu\text{g l}^{-1}$. Interference ions can generally be eliminated by using suitable masking reagents, for example, NaF solution

for Pb(II) determination in the presence of Zn(II) and Mn(II) ions [28] and sodium thiosulphate solution to eliminate Cu(II) [3].

Applications

The proposed method was applied to determine lead in drinking water samples dispensed from public drinking water coolers located in dormitory and cafeteria, Prince of Songkla University. The results show that the concentrations of Pb(II) in drinking water samples were below the detection limit of $55 \mu\text{g l}^{-1}$ (Table 1). The amounts of Pb(II) in leachates of glazed bowls were in the range of ND- $125.7 \mu\text{g l}^{-1}$ (Table 2) which is lower than that stated in FDA guideline (0.5mg l^{-1} in an acidic leaching solution).

Comparison with other Methods

The analytical performance of the developed method was compared with the previous works based on on-line preconcentration spectrophotometric technique for Pb(II) determination (Table 3). The developed method provides comparable RSD and recovery but the relative high enrichment factor with high sample throughput which is desirable for routine analysis is established. Moreover, the proposed on-line system is simpler than [3] which required multi pumping flow system and than [32] which utilized more pumps and 6-port injection valves. The only limitation might be higher detection limit but should be enough for general application. For further study, the detection limit

Table 1. Concentration of Lead in Drinking Water and % Recovery

Samples (building/site)	Pb(II) concentration ($\mu\text{g l}^{-1}$)		Recovery (%)	RSD (%)
	Added values	Measured values ^a		
Dormitory/1	0	ND	-	-
	300	292.8 \pm 0.0	97	0.4
	500	505.1 \pm 0.1	101	0.5
	700	712.0 \pm 0.0	102	0.3
Dormitory/2	0	ND	-	-
	300	275.4 \pm 0.0	92	0.4
	500	521.3 \pm 0.1	104	0.6
	700	710.2 \pm 0.1	101	0.5
Cafeteria/1	0	ND	-	-
	300	271.1 \pm 0.1	90	0.8
	500	475.7 \pm 0.3	95	2.8
	700	723.8 \pm 0.0	103	0.2
Cafeteria/2	0	ND	-	-
	300	282.6 \pm 0.0	94	0.4
	500	500.1 \pm 0.1	100	0.9
	700	668.1 \pm 0.0	95	0.3

^aValues given are the means (n = 3), ND = Non detectable (<55 $\mu\text{g l}^{-1}$).

could be improved by increasing the amount of resin in minicolumn and extending loading time so that the greater amount of Pb(II) could thus be adsorbed on the minicolumn resulting in greater sensitivity and lower detection limit.

CONCLUSIONS

The on-line preconcentration spectrophotometry as an alternative method for lead determination with the features of the simplicity of the system and instrument, cost effectiveness, high enrichment factor and short analysis time was developed. The proposed method was successfully

applied to determine lead in drinking water samples discharged from public drinking water coolers and leachates of glazed bowl samples. These applications are aimed to promote awareness and preliminary assess of toxicity of lead contaminated from the samples in our daily lives as well as to pave the way for a larger variety of samples in the future.

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Table 2. Concentration of Lead in Leachates of Glazed Bowl and %Recovery

Samples (Glazed bowl/type)	Pb(II) concentration ($\mu\text{g l}^{-1}$)		Recovery (%)	RSD (%)
	Added values	Measured values ^a		
Glazed bowl/1	0	125.7 ± 0.1	-	2.1
	300	404.7 ± 0.1	93	0.2
	500	609.4 ± 0.2	97	0.5
	700	850.3 ± 0.2	104	0.6
Glazed bowl/2	0	ND	-	-
	300	284.2 ± 0.1	98	1.1
	500	536.9 ± 0.1	107	0.6
	700	681.1 ± 0.1	97	0.6
Glazed bowl/3	0	ND	-	-
	300	291.1 ± 0.1	97	1.2
	500	533.3 ± 0.1	107	0.8
	700	683.7 ± 0.0	98	0.3

^aValues given are the means (n = 3), ND = Non detectable ($<55 \mu\text{g l}^{-1}$).

Table 3. Comparison of the Developed Method with the Previous Works Based on on-line Preconcentration Spectrophotometric Determination of Pb(II)

Adsorbent	Chelating agent	LOD ($\mu\text{g l}^{-1}$)	RSD (%)	Recovery (%)	Enrichment factor	Sample throughput (h^{-1})	Ref.
Chitosan [poly(d-glucosamine)]	Dithizone	5	4.5	95.3-108.6	8.2	15	[3]
TrisKem Pb Resin	PAR	2.7	5.4	-	-	4	[15]
Amberlite XAD-7	PAR	11	2.4	102	3	12	[31]
Carbon xerogel	Dithizone	0.9	7.8	94.25-107.02	50	4	[32]
Amberlite IR-120	PAR	55	<5	90-107	47	8	This work

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