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CHELATING RESIN AS A PRECONCENTRATION SYSTEM FOR THE DETERMINATION OF TRACE LEAD BASED ON FLOW INJECTION ANALYSIS METHOD

Aman Sentosa Panggabean[a], Subur Pasaribu[a], Muhammad Bachri Amran[b]

[a]Department of Chemistry FMIPA - University of Mulawarman, Samarinda, Indonesia-75119

[b]Division of Analytical Chemistry FMIPA - Institut Teknologi Bandung, Indonesia-40132

Abstract. Determination of trace leads by using a chelating resin packed mini-column in flow injection-preconcentration-flame atomic absorption spectrophotometric system has been done. In this research, chelating resin Polystyrene Divynilbenzene-1-(2 Pyridilazo)-2-Naphtol (PSDVB-PAN) has been used as a filler in the mini-column and the retention properties have been investigated for preconcentration of trace lead ion. Lead ion is quantitatively retained at pH 6. The optimum elution for quantitative elution occurs in 1M HNO_3. The retention capacity is 4.67 mg Pb/g resin. The analytical performance is good shown by percentage of coefficient variance was 2.24 %, the calculated detection limit was 6.71 µg L^{-1}. The method was applied to drinking water, tap water and river water for determination of lead with result percentage of recovery was >95%, indicate this technique is good to be applied for lead analysis at trace levels.

Keywords: Chelating Resin, Preconcentration, FIA, Lead(II), PSDVB-PAN.

1 Introduction

Heavy metals in the environment are generally a source of pollution. The determination of ultra trace of metals in environmental samples plays an important role in the environmental pollution monitoring, due to their toxicity. Despite the selectivity and sensitivity of analytical techniques such as atomic absorption spectrophotometer, there is a crucial need for the preconcentration of trace elements before their analysis due to their low concentrations in numerous samples (especially water samples) [1,2].

The most several methods have been proposed and used for preconcentration and separation of trace elements according to the nature of the samples, the concentrations of the analytes and the measurement techniques. Sorption preconcentration is one of the most effective methods for these metals [3]. In recent

years, chelating sorbents and ion exchangers are widely used for noble metal preconcetration. Different types of sorbents have been synthesized for the preconcentration of noble metals [4,5].

Chelating resin basically is consisted of two components that are functional of chelating group and polymer matrix as supported. Nature of from both this components will determine usage and performance from a chelating resin. The selectifity will be determined by type chelating group, while capacities, mechanic strength and the chemistry resistance determined by supporter polymer type who applied [6,7]. This means that a chelating resin with certain characteristic can be synthesis by considering both the compiler components.

The strategy determines in preconcentration to apply chelating resin is how to incorporate chelating reactant into polymer supporter material [8]. Way of simple is through impregnation technique, but chelating resin yielded generally gives unfavourable performance because chelating group which only tied in physical to earn easily escapes again at the time of its use [9,10]. To solving problems explained above, hence chelating group shall tie chemically through covalent bond at polymer applied as supporter material [11]. The existing finite, chelating resin this type has not many checked more than anything else is commercial and still be classified as fine-chemicals.

At this research, chelating resin Polystyrene Divynilbenzene-1-(2 Pyridilazo)-2-Naphtol (PSDVB-PAN) has been synthesized through an azo (-N=N-) intermediate groups, based on preliminary earlier research [12]. The retention characters is directly applicable to develop a new analytical technique based Preconcentration-Flow Injection Analysis (FIA) by exploiting it as filler material mini-column for step preconcentration and applicated to determination of trace lead in sample waters.

2 Experimental

2.1. Instrumentation and Reagents

Instrumentation. A set of on-line preconcentration system equipment constructed by peristaltic pump (Ismatec) and a chelating resin packed mini-column, atomic Absorption Spectrometer double beam GBC®-Avanta 6506 equipment, recorder system, and data analyzer Origin™ 7.0., was used for all measurements.

Reagents. All reagents were of analytical-reagent grade. A stock 1000 µg mL^{-1} of Pb(II) was prepared by dissolving 1.9008 g of $Pb(NO_3)_2$ (Merck) in aquabidest (HPLC grade, 18 MΩ) and was diluted to 1000 mL. Standard solution of Pb(II) were prepared daily by appropriated dillution of stock solution. Polystyrene Divynilbenzene-1-(2 Pyridilazo)-2-Naphtol (PSDVB-PAN) chelating resin synthesized and HNO_3 (Merck).

2.2 Preparation PSDVB-PAN packed mini-column

0.2 g PSDVB-PAN suspended in aquabidest with pH 6 was slurry packed into mini-column (2 mm i.d x 60 mm length). Before the use, the column was precondition with aquabidest adjusted to pH 6. The diagram of the on-line preconcentration system is shown in Figure 1.

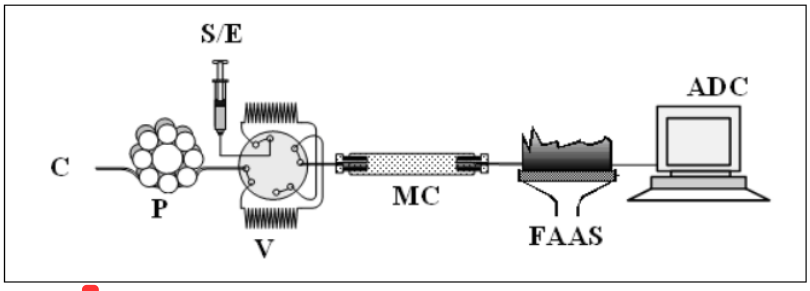

Figure 1. Schematic diagram of the Flow Injetion-Preconcentration-FAAS system. S: sample; E: eluent; C: carrier; P: peristaltic pump; V: eight-port valve; MC : PSDVB-PAN mini-column; ADC : analog digital converter

The manifold was adapted to the nebulizer system of the flame atomic absorption spectrometer (FAAS). The flow system was made up of a peristaltic pump furnished with Tygon tubes to delivery all solutions, a two-position eight-way teflon rotary valve and a PSDVB-PAN packed mini-colomn for the on-line preconcentration of lead.

2.3. Effect of the concentration of the eluent

1 mL standard of lead solution containing 10 µg L^{-1} pH 6 was passed through to mini-column at flow rate of 2 mL min^{-1}. After passage of the solution finished, the retained analyte were eluted by a 1 mL HNO_3 with concentration various 0.5-3.0 M. The eluate was taken direct to the nebulizer–burner system of an atomic absorption spectrophotometer.

2.4. Analysis of samples

For application of the proposed preconcentration method, water sample was taken in a beaker glass, and then pH of sample adjusted to 6 by 0.1M HNO_3. Water sample was passed through column at flow rate 2 mL min^{-1}. The concentration of analyte ion in the final solution were determination by FAAS at 283.3 nm.

3 Results and Discussion

3.1. Influence of pH on lead retention

pH influence at Pb(II) of retention done with batch method. Figure 2 showing pH influence chelating resin PSDVB-PAN to retention of Pb(II) ion.

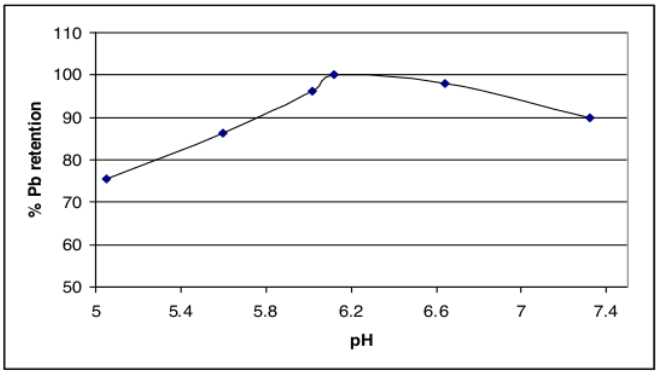

Figure 2. Influence of pH on Pb(II) retention

Pb(II) is retention maximum at pH 6.12. This thing indicates that complex formation between ligands PAN with metal Pb(II) has been reached in maximum. For research will be applied pH 6. At pH more than 6 to visible that Pb(II) amounts retention decrease resulted the resin has is saturated by Pb. Lowering of this retention value earns also resulted from existence of forming of hydroxide compound from Pb that is $Pb(OH)_2$. $Pb(OH)_2$ Compound has Ksp = 3 x 10^{-6} value is small, so that metal ion Pb is forming complex with ligand PAN decreases.

3.2. Retention capacity of PSDVB-PAN resin

One of the important fundamental value of which must be owned by a chelating resin is it's the retention capacity to certain metal ion. Determination of retention capacity of Pb(II) to chelating resin PSDVB-PAN done at optimum absorption pH with contact time during 5 minutes.

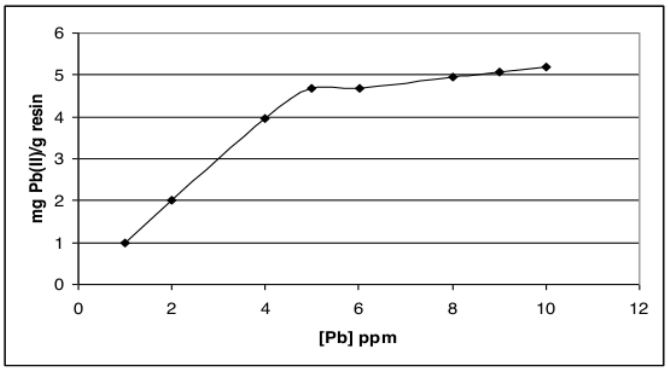

Figure 3. Retention capacity of PSDVB-PAN for Pb(II)

From result of this research, that retention capacity from synthesized chelating resin are 4.67 mg Pb(II)/g PSDVB - PAN, as seen at Figure 3. This retention capacities values would hardly determines how method preconcentration based on FIA must be done if it is applied this chelating resin as filler material of mini-column to preconcentration technique for determination of trace lead in samples.

3.3. Effect of the concentration and volume of the eluent

The elution is depend on the concentration of HNO_3 as eluent. Figure 4 shows the results for lead eluted. As shown in this figure, higher recovery was obtained when 1M or more concentrated HNO_3 was used as an eluent.

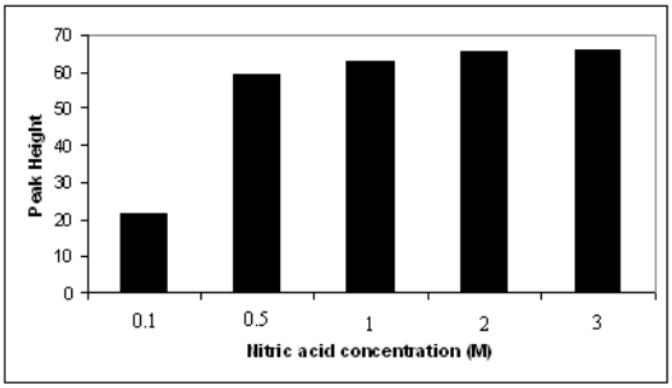

Figure 4. Signal peak height obtained as a function of concentration of eluent

The influence of the eluent volume in the step of lead desorption from the minicolumn was also investigated. Result showed that the analytical signal is constant at eluent volume within the range 1-3 mL. At volume less than 1 mL, the analytical signal considerably decreased. Figure 5 shows the influence of the eluent volume on the degree of recovery for the lead ions, 1 mL were sufficient to achieve recoveries close to 100 % in all cases.

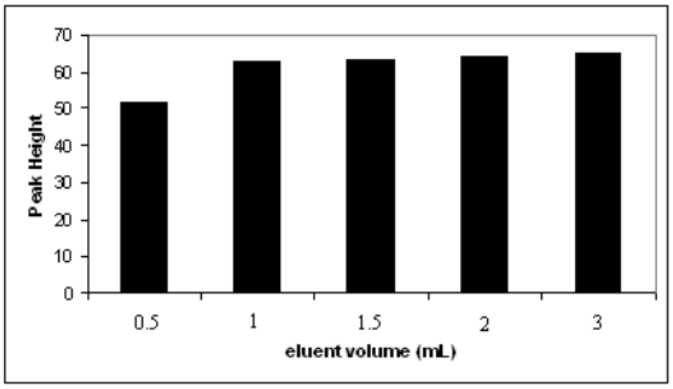

Figure 5. Signal peak height obtained as a function of volume of eluent

3.4. Effect of the sample volume

Volume based flow injection analysis was influenced by loaded sample volume. As consideration figure 6, peak height with 2 mL and 3 mL samples give no significant difference. This caused by hydrodinamic solution in sample loop that gave dillution effect. Hence, base peak of 3 mL sample become broader. Than, sample analysis was carried out with 1 mL sample. Figure 6 shown sinyal profil FI-preconcentration-FAAS.

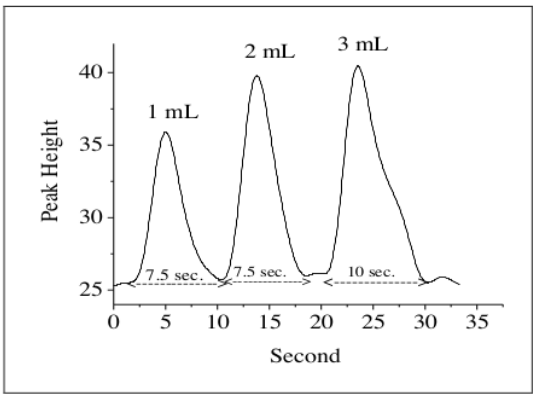

Figure 6. Signal peak height obtained as a function of standard lead 10 µg L^{-1} with various volumes loaded

3.5. Analytical Performance of the preconcentration system

The reproducibility of the preconcentration methods was evaluated by passing through mini-column of standard solution of lead 50 µgL^{-1} and repeating this procedure (n = 7). The percentage of coefficient variance (% CV) was 2.24 %.The detection limits of calculated S/N=3 was 6.71 µg L^{-1} Pb.

3.6. Determination of Lead in Water Samples

The porposed preconcentration method had been applied to water and river water for determination of lead. The results obtained are given in table 1. The recovery results indicate that proposed method can be used for lead determination in water samples.

Table 1. Results of determination of lead ions in water samples.

Samples	Pb(II), µg L^{-1}		Recovery %
	Added	Found	
	0	nd*	-
Bottled drinking water	100	99.81 ± 0.25	99.81 ± 0.03
	0	nd*	-
Ground water	100	95.73 ± 3.71	95.73 ± 0.37
	0	nd*	-
Tap water	100	97.33 ± 0.82	97.33 ± 0.08
	0	10.62 ± 1.37	-
River water	100	100.22 ± 0.76	100.22 ± 0.07

*** nd: not detected**

3 Conclusion

The proposed methods for determination of lead water and river water samples by on-line FI-preconcentration-FAAS are simple, rapid and sensitive. Polystyrene Divynilbenzene-1-(2 Pyridilazo) 2-Naphtol (PSDVB-PAN) chelating resin has been used as a mini-column filler in the step preconcentratin process. That retention property of chelating resin synthesis has been investigated for preconcentration of lead ion and eluted quantitavely by on-line, so that method can be use for lead preconcentration before determination lead by FAAS. The determination showed good analytical performance.

Acknowledgement

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AMAN SENTOSA PANGGABEAN

Department of Chemistry,

Faculty of Mathematics and Natural Sciences,

University of Mulawarman, Samarinda, Indonesia

E-mail: S304gabe@mail.chem.itb.ac.id

SUBUR P. PASARIBU

Department of Chemistry,

Faculty of Mathematics and Natural Sciences,

University of Mulawarman, Samarinda, Indonesia

E-mail: sp_pasaribu@yahoo.com

MUHAMMAD BACHRI AMRAN

Division of Analytical Chemistry,

Faculty of Mathematics and Natural Sciences,

Institut Teknologi Bandung, Indonesia

E-mail: amran@chem.itb.ac.id

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