ANALYTICAL PERFORMANCE TEST OF SILVER NANOPARTICLES CITRATE (AgNPs-Cit) IONIC FOR CHROMIUM (III) IONS DETERMINATION

BACHELOR THESIS
Submitted as a Requirement
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In the Department of Chemistry

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ABSTRACT

Chromium as heavy metal has potential the risk of air, water, and soil pollution and ultimately to the whole aquatic and terrestrial ecosystem. The objective of this research was synthesize and characterize of silver nanoparticles citrate (AgNPs-Cit) ionic by using sodium borohydride as reducing agent and trisodium citrate as a stabilizer and analyze the material performance of AgNPs-Cit ionic for Cr^{3+} determination. Material performance test consists of study the effect of pH, specific detection of Cr^{3+}, operational condition of the ratio of AgNPs-Cit:Cr^{3+}, and validation method (linear range, limit of detection (LOD), limit of quantification (LOQ), and repeatability (%RSD)). The method to synthesis of AgNPs-Cit ionic is based on chemical reduction method. Silver nanoparticles used to detect Cr^{3+} by colorimetric method.

In this study, synthesis of AgNPs-Cit ionic has been successfully done and used to detect Cr^{3+} with an indicator of color change and the absorption of Surface Plasmon Resonance (SPR) bands at specific wavelength which are the characteristic of AgNPs-Cit ionic. The AgNPs-Cit ionic give color change brownish yellow and SPR band at 399.6 nm. The color change from yellow to red also occurs when AgNPs-Cit ionic reacted with Cr^{3+} and SPR band shift from 392 to 512.5 nm. The material performance test of AgNPs-Cit ionic for Cr^{3+} determination shows good performance. pH 7 was chosen as the AgNPs-Cit ionic condition after reacting with Cr^{3+}. Specific detection shows that AgNPs-Cit ionic are only selective with Cr^{3+} and there is no effect from Cr^{6+}. Ratio (v/v) of AgNPs-Cit:Cr^{3+} 4:1 was chosen as an operational condition because it shows the best sensitivity. Under optimal conditions, the linear range was observed in the ranges concentration 0.8-3.0 ppm with LOD of 0.3 ppm and LOQ of 1.0 ppm. The correlation coefficient (R^2) of 0.9611. Repeatability shows a good precision (<5%) in 3.4% for 1.2 ppm and 1.6% for 1.6 ppm. This development method was successfully carried out with good performance so as to pave the way for further application in the real sample.

Keywords: *Chemical reduction, Colorimetric sensor, Silver nanoparticles, Metal ion detection, UV-vis spectroscopy.*

UJI PERFORMA ANALITIS NANOPARTIKEL PERAK SITRAT (AgNPs-Cit) IONIK UNTUK PENENTUAN ION CHROMIUM (III)

Syafriyanti Annur (15630032)
Program Studi Kimia, UIN Sunan Kalijaga Yogyakarta

ABSTRAK

Kromium sebagai logam berat memiliki potensi risiko polusi udara, air, dan tanah dan pada akhirnya ke seluruh ekosistem perairan dan darat. Tujuan dari penelitian ini adalah mensintesis dan mengkarakterisasi nanopartikel perak sitrat (AgNPs-Cit) ionik menggunakan natrium borohidrida sebagai zat pereduksi dan trisodium sitrat sebagai penstabil dan menganalisis kinerja material AgNPs-Cit ionik untuk penentuan Cr^{3+}. Uji kinerja material terdiri dari uji pengaruh pH, deteksi spesifik ion Cr^{3+}, kondisi operasional rasio dari AgNPs-Cit:Cr^{3+}, dan metode validasi (rentang linier, batas deteksi (LOD), batas kuantifikasi (LOQ), dan pengulangan (% RSD)). Metode untuk sintesis AgNPs-Cit ionik didasarkan pada metode reduksi kimia. Nanopartikel perak digunakan untuk mendeteksi Cr^{3+} dengan metode kolorimetri.

Berdasarkan hasil penelitian, sintesis AgNPs-Cit ionik telah berhasil dilakukan dan digunakan untuk mendeteksi Cr^{3+} dengan indikator perubahan warna dan munculnya serapan *Surface Plasmon Resonance* (SPR) pada panjang gelombang tertentu yang merupakan karakteristik dari AgNPs-Cit ionik. AgNPs-Cit ionik memberikan perubahan warna kuning kecoklatan dan pita SPR pada 399.6 nm. Perubahan warna dari kuning menjadi merah juga terjadi ketika AgNPs-Cit ionik bereaksi dengan Cr^{3+} dan pita SPR bergeser dari 392 ke 512,5 nm. Uji kinerja material AgNPs-Cit ionik untuk penentuan Cr^{3+} menunjukkan kinerja yang baik. pH 7 dipilih sebagai kondisi AgNPs-Cit setelah bereaksi dengan Cr^{3+}. Deteksi spesifik menunjukkan bahwa AgNP hanya selektif dengan Cr^{3+} dan tidak ada pengaruh dari Cr^{6+}. Rasio (v/v) dari AgNPs-Cit:Cr^{3+} 4:1 dipilih sebagai kondisi operasional karena menunjukkan sensitivitas yang paling baik. Dalam kondisi yang optimal, rentang linier diamati dalam rentang konsentrasi 0.8-3.0 ppm dengan batas deteksi 0.3 ppm dan batas kuantifikasi 1.0 ppm. Koefisien korelasi (R^2) sebesar 0.9611. Pengulangan menunjukkan presisi yang baik (<5%) dalam 3.4% untuk 1.2 ppm dan 1.6% untuk 1.6 ppm. Metode pengembangan ini berhasil dilakukan dengan kinerja material yang baik sehingga membuka jalan untuk aplikasi lebih lanjut dalam real sampel.

Kata kunci: *Reduksi kimia, Sensor kolorimetrik, Nanopartikel perak, Deteksi ion logam, Spektroskopi UV-vis.*

CERTIFICATE OF ORIGINALITY

This is to certify that:

Name	: Syafriyanti Annur
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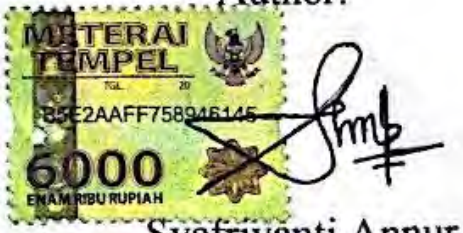
has conducted the bachelor thesis entitled:

ANALYTICAL PERFORMANCE TEST OF SILVER NANOPARTICLES CITRATE (AgNPs-Cit) FOR CHROMIUM (III) IONS DETERMINATION

which is my own opus or research. As long as I know, there is no opus or research written or published by another person except as guidance or citation in following opus grammar standardly.

Yogyakarta, 19 Agutus 2019

Author:



Syafriyanti Annur
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BACHELOR THESIS APPROVAL LETTER

About : Bachelor Thesis
Enclosure : -

To
The Dean of Faculty of Science and Technology
Sunan Kalijaga State Islamic University
Yogyakarta

Assalamu'alaikum wr.wb

After reading, checking, giving guidance, correcting and repairing, we are an advisor, have decided that a Bachelor thesis of:

Name : Syafriyanti Annur
NIM : 15630032
Title : Analytical Performance Test of Silver Nanoparticles Citrate (AgNPs-Cit) for Chromium (III) Ions Determination

have been submitted to Faculty of Science and Technology, Department of Chemistry, State Islamic University of Sunan Kalijaga of Yogyakarta as one of the requirements to get bachelor degree (S-1) in Scientific.

By those conditions, we hope the bachelor thesis can be examined as soon as possible. Thank you.

Wassalamu'alaikum wr.wb

Yogyakarta, 19 Agustus 2019
Advisor

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Letter of Agreement

This letter to declare that

Full Name	: Syafriyanti Annur
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This student has conducted a research in Student Mobility Credit Transfer Program (03rd January 2019 – 16th May 2019) with topic:

"SYNTHESIS OF SILVER NANOPARTICLE FOR COLORIMETRIC SENSOR OF CHROMIUM (III) ION"

As her Advisor, I give permission for this student to use all contents of the project results to be one of requirements for completing her bachelor degree at State Islamic University (UIN) Sunan Kalijaga Yogyakarta. I'm willing to put my name as her advisor in the undergraduate thesis.

Hat Yai, 14th May 2019
Advisor

Dr. Supunne Duangthong

CONSULTANT AGREEMENT LETTER

About : Undergraduate Thesis

To
The Dean Faculty of Science and Technology
Sunan Kalijaga State Islamic University
Yogyakarta

Assalamu'alaikum wr.wb

After reading, checking, giving guidance, correcting and repairing, we are as consultant, have decided that an undergraduate thesis of:

Name : Syafriyanti Annur
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have been correct and in accordance with the provisions as one of requirements to get undergraduate degree (S-1) in Scientific field.

Thus this letter, for the attention we say thank you.

Wassalamu'alaikum wr.wb

Yogyakarta, 16th September 2019
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CONSULTANT AGREEMENT LETTER

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Assalamu'alaikum wr.wb

After reading, checking, giving guidance, correcting and repairing, we are as consultant, have decided that an undergraduate thesis of:

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have been correct and in accordance with the provisions as one of requirements to get undergraduate degree (S-1) in Scientific field.

Thus this letter, for the attention we say thank you.

Wassalamu'alaikum wr.wb

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MOTTO

"But Allah is your protector and He is the best of helpers"
(Q. S Ali Imron: 150)

"To be intelligent isn't measured by what you know,
but how much you strive to know"
(Ms. Lisa Santos)

"Let your FAITH be bigger than your FEARS"
(Steve Jobs)

"Think as big as galaxy"
(Syafriyanti)

DEDICATION

I would like to express my sincere gratitude to
the Most Merciful God Allah SWT and also Prophet Muhammad SAW

I want to dedicate my Bachelor thesis to:

Mama,
The greatest woman that I've ever met
The sincerest heart that I've ever had

Papa,
My superhero who gave all my desires
The greatest man who gave advice and taught by example

Alma mater,
Department of Chemistry
State Islamic University of Sunan Kalijaga of Yogyakarta

PREFACE

Bismillahirrahmanirrahim

I would like to acknowledge the countless thank to the Most Merciful God who always gives His blessing and mercy in my life and there is no doubt about it. Finally, the author can complete this Bachelor thesis entitled "Analytical Performance Test of Silver Nanoparticles Citrate (AgNPs-Cit) Ionic for Chromium (III) Ions Determination".

The success of this research was supported by several institutions. I would like to thank Department of Chemistry, Faculty of Science and Technology, State Islamic University of Sunan Kalijaga who gave me the golden opportunity to take Student Receiving Exchange and Credit Transfer Scholarship in Prince of Songkla University so that I can carry out Project in Chemistry in one semester. I also thank Department of Chemistry, Faculty of Science, Prince of Songkla University for the opportunity and financial support during my research through Thailand's Education Hub for ASEAN Countries (TEH-AC) scholarships.

Personally, I would like to express my sincere gratitude to my advisor Karmanto, S.Si., M.Sc who provided me fruitful guidance, valuable comments, suggestions, and helps for completing this Bachelor thesis. I'm also grateful to Dr. Supunnee Duangthong as my advisor in PSU who gave me encouraging guidance, knowledge, and motivation during my research.

I humbly extend acknowledgments to my lab partners (Tang, Yuii, and Rung) in Analytical Laboratory CH-319, Prince of Songkla University who provided support and helps. All members of Chemistry 2015 (Kalium) who helped me throughout my journey in college. Everyone who has helped this Bachelor thesis, directly or indirectly that can't be mentioned one by one. May all of the kindness can get best in return. Finally, my greatest thanks to my dearest parents for moral and financial support and caring to encourage me to finish my study.

This Bachelor thesis describes the synthesis and characterizes of silver nanoparticles citrate (AgNPs-Cit) ionic by using sodium borohydride as reducing agent and trisodium citrate as a stabilizer and analytical performance of AgNPs-Cit ionic for Cr^{3+} determination. Synthesized of silver nanoparticles are estimated to have a good performance for Cr^{3+} determination, so the material performance test of AgNPs-Cit ionic are studied. The expected benefit is to obtain efficient synthesis method of AgNPs-Cit ionic so that it can be used to detect Cr^{3+} in real samples by colorimetric sensor easily and effectively.

In this paper, I realize there is an unintended error in writing this paper. I really allow all the readers to give their suggestion to improve in content in order to be made as one of the good examples for the next one. I also hope that my Bachelor thesis will be useful for other science researchers. *Aamiin*.

Yogyakarta, 04th August 2019

Syafriyanti Annur
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CONTENTS

LIST OF FIGURES

Page

LIST OF TABLES

Page

LIST OF APPENDIX

Page

CHAPTER I
INTRODUCTION

A. Significance of the Research

Heavy metal as raw material is extensively used in various industries to provide human needs. Heavy metal commonly used for metallurgy (a component of stainless steel, in nickel alloys), in the leather industry, electroplating, fixing pigment, and dying is Chromium. The wide usage of chromium has potential the risk of air, water, and soil pollution and ultimately to the whole aquatic and terrestrial ecosystem (Kaur, 2017). Cr as heavy metal is not biodegradable unlike organic contaminants and tend to accumulate in a living organism so that can be toxic and carcinogenic (Suresh, 2011).

Chromium exists in two oxidation states: Cr^{3+} and Cr^{6+}. Cr^{6+} is strong oxidizing agent and will react with organic compound or other reducing agents to from Cr^{3+}. Therefore, in water that is rich in organic compound, Cr^{6+} will exhibit a much shorter lifetime (USEPA, 1998). In this research, it's specific to Cr^{3+}. Chromium(III) is inorganic contaminants that found in wastewaters because it directly or indirectly discharges into the environment increasingly. Cr^{3+} ion is known to play an important role in the functioning of insulin hormone, metabolism of carbohydrates, lipids, and proteins and so the deficiency of Cr^{3+} can restrict the sugar metabolism. Based on the World Health Organization (WHO) and the United States Environmental Protection Agency (USEPA) exhibit that the maximum allowable levels of Cr^{3+} ions are 0.05 ppm and 0.1 ppm in drinking water (S. Vasudevan, 2011). Due to the monitor of Cr^{3+} concentrations, there is a need to develop a rapid, sensitive, and reliable method for selective detection of Cr^{3+}.

Based on previous research conducted by Pratiwi et al., 2013, Chromium determination was carried out by the coprecipitation method. The results showed that the coprecipitation method was less selective for Cr^{6+} ions because of the presence of Cd^{2+} ions as interference. The addition of 1 mL of Cd^{2+} 20 ppm has interfered the presence of Cr^{6+} seen from absorbance which increased by 21%. This shows that adding a small amount of Cd^{2+} will still interfere with the analysis of Cr^{6+}. Other previous researches to detect Cr^{3+} in aqueous solution have been accomplished also by various analytical tools including Atomic Absorption Spectrometry (AAS), High-Performance Liquid Chromatography (HPLC), and X-ray Fluorescence Spectrometry. The use of instruments has shortcomings such as expensive, the tools require special treatment, and require complex reagent so that its use is limited, and show the poor selectivity and sensitivity (Suresh,2018). Therefore, another method that is cheaper, practical, good selectivity and sensitivity are necessary.

In recent years, metal nanoparticles have been the focus of research because of their unique electronic, optical, mechanical and magnetic properties. One of them is silver nanoparticles because they have unusual properties compared to the state of metals in bulk or salt (Salasa, 2016). Silver nanoparticles (AgNPs) are cheaper and easy to synthesis than gold nanoparticles (AuNPs). Silver nanoparticles can detect heavy metal (Cr^{3+}) by colorimetric method. The colorimetric method is a method that utilizes visual and UV-Vis spectroscopy characterization. The visual characterization uses the naked eye to see the color change when the nanoparticle solution is reacted with Chromium. Meanwhile, on UV-Vis spectroscopy

characterization, the results are shown by changing the absorbance curve due to the addition of Cr concentration (Permana et al., 2014). This alternative colorimetric method is chosen because its utilization as a test kit for onsite detection, short-time detection, simplicity, reversibility along with high selectivity and sensitivity, which does not need any pre-treatment of sample and manual expertise, so make it a desirable technique among the researches (Kaur, 2017).

The method commonly used to synthesize metal nanoparticles, such as a chemical reduction in solution, thermal decomposition, electrochemistry, microwave, and micro emulsions. Of all these methods, the chemical reduction method is more economical than other methods because the reduction method only requires reducing agents (Zielinska et al.,2009). In addition, the chemical reduction method allows it to be produced on a large scale.

Based on previous research conducted by Elavarasi et al., 2014 synthesized of silver nanoparticles for Cr^{3+} was successfully done using chemical reduction method. Cr^{3+} determination was also done using a simple colorimetric method. The research focuses on differences in the total volume of reducing agent while the validation method was not carried out. In this research, material performance parameters are studied to get better control over the synthesis process.

This study aims to the synthesize and characterize of silver nanoparticles citrate (AgNPs-Cit) ionic by using sodium borohydride as reducing agent and trisodium citrate as a stabilizer. The citrate has been used as stabilizer or ‘capping agent’ because it has active organic groups (COOH and OH) which makes it easy to interact with chemical species and specific interaction with various metal species.

Cr^{3+} ion has high affinity to bind with AgNPs-Cit ionic to produce AgNPs aggregated structure. Other ligands such as N-(2-hydroxybenzyl)-valine, tartrate, etc. are capable to detect only single metal ion and need expensive chemicals to detect Cr^{3+} (Suresh et al., 2018). In this case, synthesized of silver nanoparticles are expected to have a good performance for Cr^{3+} determination so the material performance test of AgNPs-Cit ionic are studied. The validation method of AgNPs-cit ionic for Cr^{3+} determination is also evaluated to get a set of criteria that specify, in quantitative terms, the quality required for analytical performance to deliver laboratory test.

B. Scopes

This research is limited to:

1. AgNPs-Cit ionic synthesis process is carried out by chemical reduction method.
2. The characterization is using a simple colorimetric method.
3. The analytical performance (validation method) is limited to linearity test, the limit of detection (LOD), the limit of quantification (LOQ), and repeatability (%RSD).
4. The instrumentation is using UV-visible spectroscopy.

C. Problems Formulation

The problems formulation of this research are:

1. How to synthesize and characterize of silver nanoparticles citrate (AgNPs-Cit) ionic by using sodium borohydride as reducing agent and trisodium citrate as a stabilizer?

2. How about the material performance test of AgNPs-Cit ionic for Cr^{3+} determination?

D. Objectives

This study aims to:

1. Synthesize and characterize of silver nanoparticles citrate (AgNPs-Cit) by using sodium borohydride as reducing agent and trisodium citrate as a stabilizer.
2. Analyze the material performance of AgNPs-Cit for Cr^{3+} determination.

E. Expected Benefit

The expected benefit is to obtain efficient silver nanoparticles synthesis methods so that it can be used to detect Cr^{3+} in real samples.

CHAPTER V
CONCLUSION AND SUGGESTIONS

A. Conclusion

Based on the research that has been conducted, it can be concluded that:

1. Silver nanoparticles can be synthesis by chemical reduction method using sodium borohydride as a reducing agent, trisodium citrate as a stabilizer, and silver nitrate as a precursor marked by color change brownish yellow and SPR band at 399.6 nm which are the characteristic of AgNPs-Cit ionic.
2. The material performance test of AgNPs-Cit ionic for Cr^{3+} determination showed good performance. pH 7 was chosen as the AgNPs-Cit ionic condition after reacting with Cr^{3+}. Specific detection shows that AgNPs are only selective with Cr^{3+} and there is no effect from Cr^{6+}. Ratio (v/v) of AgNPs-Cit:Cr^{3+} 4:1 was chosen as an operational condition because it shows the best sensitivity. Under optimal conditions, the linear range was observed in the ranges concentration 0.8-3.0 ppm, detection limit of 0.3 ppm, quantification limit of 1.0 ppm, and R^2 of 0.9611. Repeatability shows a good precision (<5%) in 3.4% for 1.2 ppm and 1.6% for 1.6 ppm.

B. Suggestions

Based on the research that has been done, the author gives suggestions as follows:

1. Further research on time variations needs to be done to determine the stability of silver nanoparticles.
2. Further research needs to be tested on Cr(III) in the real sample.

3. It is necessary to study the effect of other disturbing compounds that can interfere with the detection of Cr(III).

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