

**EFFECTS OF ORGANIC SOIL AMENDMENT ON HEAVY METALS FRACTIONATION
OF TAILINGS: A CASE STUDY OF AKARA GOLD MINE, THAILAND**

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**A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Science Program in Environmental Management
(Interdisciplinary Program)
Graduate School
Chulalongkorn University
Academic Year 2011
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บทคัดย่อและแฟ้มข้อมูลฉบับเต็มของวิทยานิพนธ์ตั้งแต่ปีการศึกษา 2554 ที่ให้บริการในคลังปัญญาจุฬาฯ (CUIR)
เป็นแฟ้มข้อมูลของนิสิตเจ้าของวิทยานิพนธ์ที่ส่งผ่านทางบัณฑิตวิทยาลัย

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ผลจากการเติมวัสดุปรับปรุงดินต่อสัดส่วนรูปแบบโลหะในกากแร่ กรณีศึกษาเหมืองทองอัครา, ประเทศไทย

นางสาวลลิตา ชูณหะเชิดชัย

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต

สาขาวิชาการจัดการสิ่งแวดล้อม (สหสาขาวิชา)

บัณฑิตวิทยาลัย จุฬาลงกรณ์มหาวิทยาลัย

ปีการศึกษา 2554

ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

Thesis Title EFFECTS OF ORGANIC MATTER ON HEAVY
METAL FRACTIONATIONS OF TAILINGS : A CASE
STUDY OF AKARA GOLD MINE, THAILAND
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ลลิตา ชุณหะวัณ : ผลจากการเติมวัสดุปรับปรุงดินต่อสัดส่วนรูปแบบโลหะในกากแร่ กรณีศึกษาเหมืองทองอัครา, ประเทศไทย. (EFFECTS OF ORGANIC MATTER ON HEAVY METAL FRACTIONATIONS OF TAILINGS: A CASE STUDY OF AKARA GOLD MINE, THAILAND) อ. ที่ปรึกษาวิทยานิพนธ์หลัก: อ.ดร. ศรีเลิศ โชติพันธ์รัตน์, อ.ที่ปรึกษาวิทยานิพนธ์ร่วม: ผศ.ดร. จันทรา ทองคำเกา, 152 หน้า.

งานวิจัยนี้มีจุดมุ่งหมายเพื่อศึกษาลักษณะรูปแบบของโลหะในกากแร่ (โคบอลต์, โครเมียม, ทองแดง, แมงกานีส, นิกเกิล, ตะกั่ว และสังกะสี) และศึกษาการชะของโลหะของกากแร่ผสมกับกรดอะซิติกซึ่งใช้เป็นวัสดุปรับปรุงดินในอัตราส่วนและเวลาที่แตกต่างกัน ซึ่งทำการเก็บตัวอย่างจำนวน 10 จุด จากพื้นที่ที่ทิ้งกากแร่ในบริเวณเหมืองทองอัครา จังหวัดพิจิตร ประเทศไทย โดยนำกากแร่มาวิเคราะห์ด้วยวิธีการสกัดลำดับขั้น หรือ BCR Community Bureau of Reference ทั้งหมด 3 ขั้นตอนและการย่อยกากสุดท้ายด้วยกรดเข้มข้นโดยใช้เครื่องไมโครเวฟในรูปปริมาณรวม โดยวิธีการย่อยด้วยกรดเข้มข้น (aqua regia) และในรูปแบบที่สามารถดูดซึมได้ โดยวิธีสกัดลำดับขั้นคือ ขั้นที่หนึ่ง สองและสาม (the Standards, Measurements and Testing Programme, SM&T) และวิเคราะห์หาปริมาณความเข้มข้นของโลหะต่างๆให้อยู่ในหน่วยมิลลิกรัมต่อกิโลกรัมดิน โดยใช้เครื่อง Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES)

ผลการศึกษากากแร่พบว่า ที่การสกัดลำดับขั้นที่หนึ่ง (BCR1) มีร้อยละของการชะของโลหะเทียบกับค่าความเข้มข้นของการชะโลหะทุกลำดับขั้นของโลหะนั้นเรียงลำดับจากมากไปน้อยเป็นดังนี้ แมงกานีส โคบอลต์ นิกเกิล ทองแดง สังกะสี ตะกั่วและโครเมียม โดยมีค่าเท่ากับ 32.29% (622.91มก./กก.ของดิน) 8.88% (2.08มก./กก.ของดิน) 8.20% (7.68มก./กก.ของดิน) 8.11% (7.68มก./กก.ของดิน) 6.80% (16.10มก./กก.ของดิน) 3.85% (3.74มก./กก.ของดิน) และ 0.45% (0.55 มก./กก.ของดิน) ตามลำดับ และที่การสกัดลำดับขั้นที่สอง (BCR2) มีร้อยละของการชะของโลหะเทียบกับค่าความเข้มข้นของการชะโลหะทุกลำดับขั้นของโลหะเรียงลำดับจากมากไปน้อยเป็นดังนี้ โคบอลต์ แมงกานีส ตะกั่ว นิกเกิล สังกะสี โครเมียมและทองแดง โดยมีค่าเท่ากับ 51.90% (15.95มก./กก.ของดิน) 40.82% (787.47มก./กก.ของดิน) 34.91% (33.94มก./กก.ของดิน) 26.40% (6.71มก./กก.ของดิน) 22.62% (53.58มก./กก.ของดิน) 13.27% (16.46มก./กก.ของดิน) และ 12.33% (11.67 มิลลิกรัมต่อกิโลกรัมดิน) ตามลำดับ เมื่อพิจารณาจากร้อยละการชะของธาตุที่ลำดับขั้นที่หนึ่งและสองรวมเรียงจากน้อยไปมากเป็นดังนี้ แมงกานีส โคบอลต์ ตะกั่ว นิกเกิล สังกะสี ทองแดงและโครเมียม ตามลำดับ

เมื่อนำกากแร่ดินผสมกับกรดอะซิติกทำเป็นวัสดุปรับปรุงดินในอัตราส่วนและเวลาที่แตกต่างกัน โดยมีปริมาณเท่ากับ 5% 7% และ 10%ของปริมาณอินทรีย์วัตถุในดิน และนำไปบ่มทิ้งไว้ 1 7 15 และ 30 วัน ตามลำดับ ซึ่งในตัวอย่างกากแร่ มีพหุคูณเท่ากับ 8.25 และกรดอะซิติกมีปริมาณอินทรีย์วัตถุเท่ากับร้อยละ 74.59 เมื่อนำมาผสมให้ได้ปริมาณอินทรีย์วัตถุตามร้อยละข้างต้นและนำไปศึกษาผลของปริมาณของวัสดุปรับปรุงดินและเวลาที่แตกต่างกันพบว่าเมื่อนำผลไปวิเคราะห์ความแปรปรวนแบบจำแนกทางเดียว (ONE-WAY ANOVA) และการทดสอบ Post hoc โดยใช้เทคนิควิธีกำลังสองน้อยที่สุด ที่อัตราส่วนของปริมาณสารอินทรีย์ในดินเท่ากับร้อยละ 10 นั้นจะทำให้โลหะที่ศึกษาส่วนใหญ่มีการชะออกมาน้อยที่สุดและมีความแตกต่างอย่างมีนัยสำคัญเมื่อเทียบกับอัตราส่วนอื่นๆและกลุ่มควบคุม และเมื่อนำผลทั้งหมดมาวิเคราะห์หาความสัมพันธ์ระหว่างเวลาและสัดส่วนของวัสดุปรับปรุงดินที่ต่างกันด้วยการวิเคราะห์สมการถดถอยเชิงเส้นกำลังสอง โดยพิจารณาที่วิธีสกัดลำดับขั้นแรกเป็นหลัก พบว่าเมื่อมีการเพิ่มขึ้นของปริมาณอินทรีย์วัตถุที่ลำดับขั้นที่หนึ่ง (BCR1) อัตราการชะของ ทองแดง สังกะสี และตะกั่ว ลดลง และที่วิธีสกัดลำดับขั้นที่สอง (BCR2) อัตราการชะของทองแดง โครเมียม และตะกั่วลดลง พิจารณาที่ค่าสัมประสิทธิ์สหสัมพันธ์สูงกว่า 0.5 และเมื่อพิจารณาที่เวลาการบ่มนานขึ้นพบว่าที่ลำดับขั้นที่หนึ่ง (BCR1) อัตราการชะของทองแดง และตะกั่ว ลดลง และที่วิธีสกัดลำดับขั้นที่สอง (BCR2) พบว่าอัตราการชะของนิกเกิลลดลงโดยพิจารณาที่ค่าสัมประสิทธิ์สหสัมพันธ์สูงกว่า 0.5 เช่นกัน

สาขาวิชา.....การจัดการสิ่งแวดล้อม.....

ลายมือชื่อนิสิต.....

ปีการศึกษา.....2554.....

ลายมือชื่อ.ที่ปรึกษาวิทยานิพนธ์หลัก.....

ลายมือชื่อ.ที่ปรึกษาวิทยานิพนธ์ร่วม.....

5287562320 : MAJOR ENVIRONMENTAL MANAGEMENT

KEYWORDS: BCR SEQUENTIAL EXTRACTION / TAILINGS / ORGANIC SOIL AMENDMENT / HUMIC ACID / STATISTIC ANALYSIS

LALITA CHUNHACHERDCHAI: EFFECTS ON ORGANIC MATTER ON HEAVY METAL FRACTIONATIONS OF TAILINGS: A CASE STUDY OF AKARA GOLD MINE, THAILAND. ADVISOR: SRILERT CHOTPANTARAT, Ph.D., COADVISOR: ASST. PROF. CHANTRA TONGCUMPOU, Ph.D., 152 pp.

The purpose of this study was to investigate metal fractionations of mine tailings (i.e., Co, Cr, Cu, Mn, Ni, Pb and Zn) and study of metal leaching from mixtures of tailing and humic acid in different incubation periods and various organic matter ratios. Ten tailing samples were taken from tailings storage facilities of Akara gold mine in Pichit province. Samples were determined using the three-step modified BCR sequential extraction (i.e., BCR1, BCR2 and BCR3) and the additional step of strong acid digestion or aqua regia for residual fraction and analyzed all fractions by Inductively Couple Plasma-Optical Emission Spectrometer (ICP-OES) and reported their concentrations in the unit of mg/kg soil. Amount of metals in such tailings were showed in percentage of BCR1, derived from concentrations of each metal in BCR1 divided by those in all fractions, and were ranged in descending order as follows: Mn > Co > Ni > Cu > Zn > Pb > Cr, that were 32.29% (622.91 mg/kg soil), 8.88% (2.08 mg/kg soil), 8.20% (7.68 mg/kg soil), 8.11% (7.68 mg/kg soil), 6.80% (16.10 mg/kg soil) , 3.85% (3.74 mg/kg soil) and 0.45% (0.55 mg/kg soil), respectively. Percentage of metal in BCR 2 were ranged in descending order as follows: Co > Mn > Pb > Ni > Zn > Cr > Cu , that were 51.90% (15.95 mg/kg soil), 40.82% (787.47 mg/kg soil) , 34.91% (33.94 mg/kg soil), 26.40% (6.71 mg/kg soil), 22.62% (53.58 mg/kg soil), 13.27% (16.46 mg/kg soil) and 12.33% (11.67 mg/kg soil), respectively. As a result, the mobility orders of these metals were Mn > Co > Pb > Ni > Zn > Cu > Cr.

Tailings were mixed with humic acid with various ratios of organic matter (5%, 7% and 10%) and with different incubation periods (1, 7, 15 and 30 days) in order to determine the optimum condition of different organic amendment ratios and times on reduction of metal mobility. Soil tailings had a pH around 8.25 and humic acid were 74.59% organic matter. When samples were mixed into those ratios and then analyzed all results through SPSS Statistic program by using ONE-WAY ANOVA and post hoc test with LSD method (Fisher's Least significant difference). The optimum condition of mixing were ten percentage of organic matter as compared to other treatments, which showed statistical significantly lower concentration of metals leached out ($p < 0.05$). Semi-log linear regression analysis was then used to determine the relationship between times and ratios of organic matter with metal concentrations in each fraction. Metal concentrations of first two fraction were mainly focused on and found that amounts of Cu Zn and Pb were reduced in BCR1 and Cu Cr and Pb were reduced in BCR2 ($R^2 > 0.5$) . Moreover, the results showed that amounts of Cu and Pb in BCR 1 and Ni in BCR2 reduced with increasing time aging increased ($R^2 > 0.5$).

Field of Study: Environmental Management

Student's Signature:.....

Academic Year:..... 2011

Advisor's Signature:.....

Co-advisor's Signature:.....

ACKNOWLEDGEMENTS

Firstly, I would like to express my deepest gratitude to my thesis advisor, Dr. Srilert Chotpantararat, for his valuable suggestions, huge motivations, and kind guidance throughout the thesis work. I would like to sincerely thank to my co-advisor Assistant Prof. Dr. Chantra Tongcumpou, who give suggestion that stimulate the advance of this work. In addition, I would like to thank the thesis committee chairperson, Assistant Prof. Dr. Ekawan Luepromchai, and all thesis committee members; Associate Prof. Dr. Wasan Pongsapich and Dr. Benjaporn Boonchayaanant, for their reviews, helpful suggestions, and strong comments.

Particularly, I also would like to thank the EHWL laboratory officers, Miss Chantana Intim and Miss Benjawan Noinumnon for helping and giving advices related to laboratory equipment especially, ICP-OES in deepest details. Miss Saowaros Mornsuparp, a librarian, for welcome and warm supports. Moreover, I also thank Mr. Kamphol Pantakua, a researcher of Thailand Development Research Institute (TDRI), for his great help in the Statistic fields throughout this study. Mr. Sorn Simatrang, a master degree student of Case Western Reserve University, who send an important chemical substance from USA to Thailand in a short notice, and his kindness for download some online journals and books references which cannot be download in Thailand. Miss Pensiri Akkajit and Mr Mongkolchai Assawadithalerd Ph.D. students at International Postgraduate Program in Environmental Management, Graduate school, Chulalongkorn University for their great help in field and laboratory works that related to this work. Mr. Songsak Haesakul, a master degree student in Faculty of Information Technology, King's Mongkut University of Technology North Bangkok for his great help in some accessories. Furthermore, all of my friends and colleagues whose name not to be mentioned but I keep in mind and feel thankful of them.

Importantly, I would like to sincerely thank for funding supported by International Postgraduate Programs in Environmental Management, Graduate school Grant and Faculty of Science Funding of Chulalongkorn University for supporting my study. I also thank Akara Gold Mine for providing all soil tailings materials.

Finally, I feel proud to dedicate this thesis with all respects to my beloved family for their endless love, encouragement, and inspiration throughout my entire study.

CONTENTS

	Page
Abstract (Thai).....	iv
Abstract (English).....	v
Acknowledgements.....	vi
Contents.....	vii
List of Tables.....	xi
List of Figures.....	xiii
List of Abbreviations.....	xvii

CHAPTER

I	INTRODUCTION.....	1
	1.1 General Statement.....	1
	1.2 Objectives.....	5
	1.3 Hypotheses.....	6
	1.4 Scope of Work.....	6
	1.5 Benefit of this study.....	8
II	LITERATURE REVIEW AND THEORETICAL BACKGROUND.....	9
	2.1 Mining characteristics and systems.....	9
	2.2 Sequential extraction	10
	2.3 Bioavailability.....	11
	2.4 Soil parameters	13
	2.4.1 Soil pH.....	13

	Page
CHAPTER	
2.4.2 Organic matter.....	13
2.5 The presence of heavy metals.....	14
2.6 Seven interested metals.....	15
2.6.1 Cobalt (Co).....	15
2.6.2 Copper (Cu).....	16
2.6.3 Chromium (Cr).....	16
2.6.4 Manganese(Mn).....	17
2.6.5 Nickel (Ni).....	18
2.6.6 Lead (Pb).....	18
2.6.7 Zinc (Zn).....	19
III METHODOLOGY.....	20
3.1 Introduction.....	20
3.2 Study Sites.....	21
3.3 Soil tailings sample collection and preparation.....	21
3.3.1 Soil sample collection and preparation.....	21
3.3.2 Preparing soil samples into soil amendment.....	21
3.4 Analysis parameter.....	22

	Page
CHAPTER	
3.4.1 Soil Chemical properties	22
3.4.1 organic matter.....	22
3.4.2 calculating soil organic amendment (%SOM)...	23
3.5 Metal analysis.....	23
3.5.1 BCR sequential extraction	23
3.5.2 Total digestion (EPA3052).....	25
3.6 Soil amendment analysis.....	26
3.7 Quality control.....	26
3.8 Data analysis	26
IV RESULTS AND DISCUSSION.....	27
4.1 The Physicochemical characteristic of soil and humic acid..	27
4.2 Total metal concentrations in soil tailings.....	28
4.3 Validation of the method.....	35
4.4 Comparison between aging time and organic amendment ratio.....	35
4.4.1 Cobalt.....	38
4.4.2 Copper	42

	Page
CHAPTER	
4.4.3 Chromium.....	47
4.4.4 Manganese.....	52
4.4.5 Nickel.....	57
4.4.6 Lead	61
4.4.7 Zinc.....	65
4.5 Fractionation patterns of metals	69
V CONCLUSIONS AND RECOMMENDATIONS.....	72
5.1 Conclusions.....	72
5.2 Recommendations.....	73
REFERENCES.....	74
APPENDICES.....	79
APPENDIX A	80
APPENDIX B	81
APPENDIX C	82
APPENDIX D	83

	Page
CHAPTER	
APPENDIX E.....	94
APPENDIX F.....	142
BIOGRAPHY.....	152

List of Tables

	Page
Tables	
2.1 The three-step BCR sequential extraction scheme.....	12
4.1 Physicochemical parameter of Soil tailings and Humic acid.....	27
4.3 Average percentage proportion of tailing from ten locations in BCR1 and BCR2 from maximum to minimum.....	35
4.4 Metal concentrations (mg/kg) of Certified and measured reference material (CRM 025-050).....	35
4.4 Average all elements concentrations (mg/kg soil) in 1, 7, 15 and 30 days in all fraction.....	37
4.4 Average all elements concentrations (mg/kg soil) in 1, 7, 15 and 30 days in all fraction (Cont.).....	38
4.6 All equations in each metal and fractions from Regression analysis.....	70
4.7 Ranking of influence of SOM amount and time aging on reduction of heavy metal leaching.....	71

List of Figures

Page

Figures

1.1	Sketch map of Akara Gold Mine along the boundary of Phichit and Petchaboon provinces, showing tailing storage facility(TSF) and other land utilities.....	3
2.1	Mine/mill tailings environment simplistic system model.....	10
2.2	Sketch of a soil organic matter (SOM) showing the various kinds of functional group associated with such soil material	13
3.1	Experimental design in this study	20
4.1	Ten locations in Tailing Storage Facilities, TSF.....	28
4.2	The TSF in Akara gold mine.....	28
4.3	How to sample soil tailings samples from TSF area in each punch.....	28
4.4	Average Co concentration of all fraction from ten locations.....	31
4.5	Average Cr concentration of all fraction from ten locations	31
4.6	Average Cu concentration of all fraction from ten locations.....	32
4.7	Average Mn concentration of all fraction from ten locations	32
4.8	Average Ni concentrations of all fraction from ten locations	33
4.9	Average Pb concentration of all fraction from ten locations	33
4.10	Average Zn concentration of all fraction from ten locations	34
4.11	Averages Co concentrations in BCR1 within 30 days at different percentages of SOM.	41
4.12	Averages Co concentrations in BCR2 within 30 days at different percentages of SOM.	41
4.13	Averages Co concentrations in BCR3 within 30 days at different percentages of SOM.	42
4.14	Averages Co concentrations in residual fraction within 30 days at different percentages of SOM.	42
4.15	All fraction percentages of Co within 30 days at different percentages of SOM.	43

Figures

4.16	Averages Cr concentrations in BCR1 within 30 days at different percentages of SOM.	45
4.17	Averages Cr concentrations in BCR2 within 30 days at different percentages of SOM.	45
4.18	Averages Cr concentrations in BCR3 within 30 days at different percentages of SOM.	46
4.19	Averages Cr concentrations in Residual fraction within 30 days at different percentages of SOM.	46
4.20	All fraction percentages of Cr within 30 days at different percentages of SOM.	47
4.21	Average Cu concentrations in BCR1 within 30 days at different percentages of SOM.	50
4.22	Average Cu concentrations in BCR2 within 30 days at different percentages of SOM.	50
4.23	Average Cu concentrations in BCR3 within 30 days at different percentages of SOM.	51
4.24	Average Cu concentrations in residual fraction within 30 days at different percentages of SOM.	51
4.25	Percentages of Cu in all fractions within 30 days at different percentages of SOM.	52
4.26	Average Mn concentrations in BCR1 within 30 days at different percentages of SOM.	54
4.27	Average Mn concentrations in BCR2 within 30 days at different percentages of SOM.	55
4.28	Averages Mn concentrations in BCR3 within 30 days at different percentages of SOM.	55

Figures

4.29	Averages Mn concentrations in Residual fraction within 30 days at different percentages of SOM.	56
4.30	All fraction percentages of Cr within 30 days at different percentages of SOM.	56
4.31	Average Ni concentrations in BCR1 within 30 days at different percentages of SOM.	58
4.32	Average Ni concentrations in BCR2 within 30 days at different percentages of SOM.	59
4.33	Average Ni concentrations in BCR3 within 30 days at different percentages of SOM.	59
4.34	Averages Ni concentrations in Residual fraction within 30 days at different percentages of SOM.	60
4.35	All fraction percentages of Ni within 30 days at different percentages of SOM.	60
4.36	Average Pb concentrations in BCR1 within 30 days at different percentages of SOM.	63
4.37	Average Pb concentrations in BCR2 within 30 days at different percentages of SOM.	63
4.38	Average Pb concentrations in BCR3 within 30 days at different percentages of SOM.	64
4.39	Average Pb concentrations in residual fraction within 30 days at different percentages of SOM.	64
4.40	Percentages of Pb in all fractions within 30 days at different percentages of SOM.	65
4.41	Average Zn concentrations in BCR1 within 30 days at different percentages of SOM.	67

Figures

4.42	Average Zn concentrations in BCR2 within 30 days at different percentages of SOM.	67
4.43	Average Zn concentrations in BCR3 within 30 days at different percentages of SOM.	68
4.44	Average Zn concentrations in residual fraction within 30 days at different percentages of SOM.	68
4.45	Percentages of Zn in all fractions within 30 days at different percentages of SOM.	69

LIST OF ABBREVIATIONS

°C	Degree Celsius
AMD	Acid Mine Drainage
BCR	Community Bureau of Reference
BCR1	Exchangeable Fraction
BCR2	Reducible Fraction
BCR3	Oxidisable Fraction
Res	Residual Fraction
CRM	Certified Reference Material
g	Gram
g/kg	Gram per kilogram
GPS	Global Positioning System
HCl	Hydrochloric Acid
HNO ₃	Nitric Acid
H ₂ O ₂	Hydrogen peroxide
ICP-OES	Inductively Coupled Plasma-Optical Emission Spectrometers
L	Liter
M	Molar
mg/dm ³	milligram per cubic decimeter
mg/kg	Milligram per kilogram
mL	Milliliter
mol/L	Mole per liter
mV	Millivolt
OM	Organic Matter
TSF	Tailings Storage Facilities
rpm	Round per minute

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g/kg	Gram per kilogram
GPS	Global Positioning System
HCl	Hydrochloric Acid
HNO ₃	Nitric Acid
H ₂ O ₂	Hydrogen peroxide
ICP-OES	Inductively Coupled Plasma-Optical Emission Spectrometers
L	Liter
M	Molar
mg/dm ³	milligram per cubic decimeter
mg/kg	Milligram per kilogram
mL	Milliliter
mol/L	Mole per liter
mV	Millivolt
OM	Organic Matter
TSF	Tailings Storage Facilities
rpm	Round per minute

CHAPTER I

INTRODUCTION

1.1 General Statement

Mining industry becomes one of environmental problems for human health because it produces waste materials of variable nature and extent. Mining processes include raw material grinding, ore refining and solid waste disposal which can be a contaminated sources to the environment (Adriano et al., 2004). The mining and smelting of non-ferrous metals has caused soil pollution, metal dusts emanation, water, effluents and seepage (Alloway, 1990). The end product residue from mining industry is called “tailings” and almost of tailings produced in the world are pumped into large surface impoundment known as “tailings dams”(Lottermoser, 2003) or tailings storage facilities (TSF) (Akara, 1999). Many research studies have shown the adverse impacts resulted from mining industries. For examples, pollution of mining industries in Brazil generated from gold-mining activities in which mercury compound was used in the process for concentrating the gold; consequently, high mercury concentrations have been found in fish and resulted to a long-lasting effect onto metal cycling mechanism in the tropical forest ecosystem (Salomon, 1995). Another mining problem was found at the San Quintín mine area in Spain, after the San Quintín mine was definitely closed in 1988; heavy metals contaminated (Pb, Zn, and Ag) sludge from floating process of the low grade mineral heavy metals such as was distribute as sediment cover a large area and resulted to great impact (Rodríguez et al., 2009). In Mexico, Méndez et al., (2003) found arsenic contamination of groundwater which has been suspected that sources of As came from both natural and anthropogenic sources in Zimapán, which had been operated more than 60 years. After ceased operation of Gold, In the mid 1970s, in Central Rand, South Africa more than 2,400 tons gold had been mined, generated huge of solid waste from mining and metallurgy process. Consequently, massive of dumped tailing waste from of gold mining industries was in the area have remained undisturbed for almost a century, and

exposed to oxygenated rainwater (Cukrowska, 2001). Furthermore, in England and Wales, the area around 20,000 contaminated sites were found and needed to be remediated in order to reduce the risk to the environment and human health (Environmental Agency, 2004).

For Thailand, Akara Gold Mine is the largest gold mine, located at the boundary of the two provinces; Pichit and Petchaboon (Fig.1). This mine has been operated for ten years. During the gold extraction process, waste rocks are burst and partly used in a tailings storage facility (TSF) construction. During ore processing, Akara Gold Mine produces approximately 750,000 dry tons of tailings per year, and these tailings are deposited in the TSF. The TSF locates in the southern area of the mine (Fig.1) (Changul et al, 2010a). Several studies related to mining have been conducted in this area, Chotpantararat et al., (2008) reveal that heavy metal concentration in groundwater collected from shallow and deep wells at the mining site over two-year period and showed a relatively high concentration of manganese, supporting by the laboratory results of the batch and column desorption experiments. Changul et al., (2010a, 2010b) investigated chemical characteristics and acid drainage assessment of mine tailing including the acidic potential of waste rocks. The results shows that Acid Mine Drainage (AMD) generation may possibly occur a long time after mine closure due to the dissolution lag time of acid-neutralizing sources. Some waste rocks acidic generation may occur in the future based on environmental conditions, particularly the oxidation of sulfide minerals by the combination of oxygen and water. Furthermore, tailings leachates contain heavy metals exceeding the standards at low pH. As mentioned, under AMD condition, TSF would be a major contaminant factor in the future.

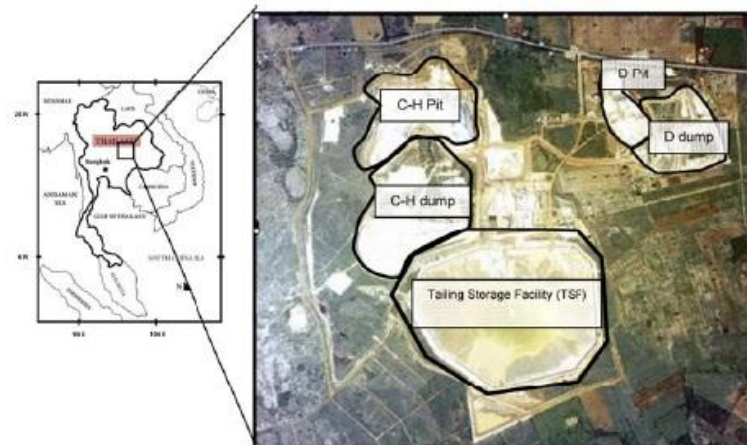


Figure 1.1 Sketch map of Akara Gold Mine along the boundary of Phichit and Petchaboon provinces, showing tailing storage facility(TSF) and other land utilities (map modified from Changul et al., 2010a)

To assess the mobility, sorption, and transportation of contaminants in the natural environment in order to understand their environmental impact, several approaches are needed for examining. Batch or column leaching experiments have received much attention. BCR sequential extraction is one of the methods to understand the fate and speciation of metals which indicates and hence leads to a better basis for site risk assessment (Cukrowska, 2004 and Iwegbue et al., 2007). In Iran, Jalali and Knablari (2008) studied the aging effect on the distribution of calcareous soil by using seven conditions (ranging in time from 3 hours to 28 days) and five-step sequential extraction was carried out in their experiments. They found that in the first three hours, metals in the exchangeable fraction were increased and slowly decreased as time passed. Brunori et al. (2005) investigated the kinetic fractionation method (collecting samples in each 15, 30, 60, 100, 150, 240, 360, and 960 minutes) and found that the addition of red mud into contaminated soil directly decreased the mobility of heavy metals from soil. Lu et al. (2005) also revealed that aging of soil affects the fractionation of heavy metals. Their experiments were carried out by spiking a total of 500 mg/kg of Cu, Zn, Pb and 2.5 mg/kg Cd to three tropical Chinese soils and analyzed according to the fractionation of the metals according to the aging time. They found that metals in the exchangeable fraction were increased in the first three hours and then as time

passed metals transformed slowly into other fraction. Bacon and Davidson (2008); Akkajit and Tongcumpou (2010) found that the first two steps of BCR are the most mobile and also increase bioavailability, which has a major risk on environmental impacts. Janos (2010) used many inorganic and organic amendment types to compare mobility and bioavailability the leachability of cadmium: the most effective from his comparison is humate K. Among many tests suggested for various purposes, a three steps procedure developed in EC standards, Measurement and Testing Programme (so call BCR test) became more popular, as it achieved a high degree of standardization and harmonization (Quevauviller, 1998; Rauret et al., 1999; Bacon and Davidson, 2008).

Pollutants consist of inorganic and organic chemicals. The Brønsted-Lowry concepts of acids and bases were used to describe the various reaction and interaction occurring in a soil water pollutant system. Chemical reactions in the pore water include (1) acid-base reactions and hydrolysis (2) oxidation-reduction (redox) reactions and (3) speciation and complexations (Taylor& Francis group, 2007).

Different soil contains organic matter which may be different in characteristics and properties. Colloidal soil organic matter has a major influence on the chemical properties of soils, and can be divided into non-humic and humic substances. They are formed by secondary synthesis reactions involving microorganisms and have characteristics which are dissimilar to any compounds in living organisms (Alloway, 1995). Organic matter is very important in the transportation and accumulation of metallic ions (Kabata Pendias and Pendias, 2001), which affect micronutrient availability (Quevauviller, 2002), due to its performance as an essential sorbent of plant macronutrients, micronutrients, heavy metal cations (particularly of copper and manganese) (Sparks, 1995) and organic material. All reactions lead to the formation of water-soluble and/or water-insoluble complexes (Kabata Pendias and Pendias, 2001). Furthermore, Dermont (2008) found that an increase in the soil pH or organic matter contents inhibited the metal's extractability and mobility.

The tailing from mining sites is one of a major concern for mining management because TSF requires an extremely large space and appropriate take care. Since most of tailing waste from mining contains several heavy metals, leachability of the heavy metal can generate an environmental problem. Comparable to heavy metals contaminated soil, an addition of organic matter into the soil has been widely used for leachability prevention or immobilization the metals in soil. Their mobility in soil system depends on their chemical forms, the nature of the organic matter and interaction among soil components (Gadepalle et al., 2007; Harter &Naidu, 1995; Janos et al., 2010). Egli et al., (1999) studied in lead, cadmium and Zinc contribution and found that major and minor chemical component behaviors were mainly determined by decomposition of organic matter. In soil remediation, soil amendment can be used alone or more often in a mixture with other amendments (Brunori, 2005 and Janos, 2010). The similar approach was introduced in this study. Organic matter was added into the mine tailing in order to investigate the possibility of its effect on mobility of the metals.

Therefore, the objective of this study is to determine the effects of organic matter and tailings ratios as well as aging time on fractionation of heavy metals in soil tailings from tailings storage facilities (TSF) of Akara Gold Mine, Pichit province. Three-step modified BCR sequential extraction was the method used to determine mobile fraction of metals.

1.2 Objectives

The main purpose of this study is to determine the effect of organic matter addition on fractionation of heavy metals in tailings from tailings storage facilities (TSF) of Akara Gold mining site in Pichit province. Two sub-objectives of this study are shown below:

1.2.1 To determine the effect on metal fractionation at different ratio of organic matter and tailing investigate heavy metals in tailings

1.2.2 To study the effect of aging time and tailing at different ratio on metal fractionation.

1.3 Hypothesis

Different amount of organic matters addition into tailing and aging period can be affected on heavy metals fractionation.

1.4 Scope of Work

1. The sampling sites located in TSF area, Akara gold mine, Pichit provinces. Samples were collected in ten sampling spots at one meter depth in April, 2010.

2. Organic Matter used in this study is humic acid.

3. Soil properties: all tailings samples and humic acid were analyzed

for:

3.1 Soil pH (1:2 soil/water suspensions) by pH meter

3.2 Soil organic matter content (OM): by wet digestion according to the Walkley-Black procedure (Benton Jones, 2001)

4. Seven metals of interest in this study include:

- Cobalt (Co)
- Chromium (Cr)
- Copper (Cu)
- Manganese (Mn)
- Nickel (Ni)

- Lead (Pb)
- Zinc (Zn)

5. Metal Analysis: concentration of each elements (Co, Cr, Cu, Mn, Ni, Pb, and Zn) were analyzed by Inductively Coupled Plasma-Optical Emission Spectrometers (ICP-OES).

5.1 Soil tailings samples

Soil samples were analyzed for total digestion according to EPA standard method (EPA-Method 3052, 1996) and the three step of BCR sequential extraction procedure developed by the Standards, Measurements and Testing programme, SM&M. The supernatant sample in each fractionation step was analyzed.

5.2 Humic acid samples

Humic acid, analytical grade, were analyzed for total digestion according to EPA standard method (EPA-Method 3052, 1996) and the three step of BCR sequential extraction procedure developed by the Standards, Measurements and Testing programme, SM&M. Supernatant from each fractionation step were analyzed as same as soil tailings samples.

5.3 Soil amendment sample

Mixture of tailings sample and humic acid were calculated into propotion of different percentage of Organic matter (OM) and Time were inoculated and collected.

6. For the accuracy of total digestion, Certified Reference Material (CRM 025-050) was used to determine metals in the supernatant.

7. Statistical Analysis

The concentration data set of the elements (Co, Cr, Cu, Mn, Ni, Pb and Zn) was analyzed by using the select cases, One-Way ANOVA, Statistic program to determine the correlation of bioavailability of metal in each fraction, and used for explain relation between Time, OM and elements (Co, Cr, Cu, Mn, Ni, Pb and Zn).

1.5 Benefit of this study

At the end of the study, the selected soil tailings and humic acid background properties (soil pH, organic matter content (OM) and the relationship between metals (Co, Cr, Cu, Mn, Ni, Pb and Zn) would have been investigated. The total digestion and the three-step BCR sequential extraction procedure recommended by the Standards, Measurements and Testing Programme of the European Union (SM&T) were used to determine the metal contents in total and available forms, respectively. Moreover, the best soil amendment condition was chosen for further research.

Furthermore, this study can be assessed or developed for further appropriate conditions in order to reduce heavy metals released to environment. Moreover, the results may be an recommendation for Akara Gold Mine to apply for tailings management

CHAPTER II

THEORETICAL BACKGROUND AND LITERATURE REVIEWS

2.1 Mining characteristics and system

Mining is the first operation of a mineral or energy resources in the commercial exploitation. Mining industries have many operations such as mining, mineral processing, and metallurgical extraction. Obtaining components of mined material depends on the extraction of material from the ground. The aim of mineral processing or beneficiation is; to physically separate and, to concentrate the ore minerals. On the other hand, the goal of metallurgical extraction is; to destroy the crystallographic bonds in the ore mineral in order to recover the source of element or compound. Mining is always associated with mineral processing such as crushing, grinding, gravity, magnetic or electrostatic separation and floatation. Sometimes, it is dealt with the metallurgical extraction of commodities such as gold, copper, nickel, uranium or phosphate (such as heap leaching; vat leaching; in situ leaching). All three operations of mining industry –mining, mineral processing and metallurgical extraction- produced waste is called “Mine Wastes” that defined as solid, liquid, or gaseous by-products of these activities. They are unwanted, have no current economic value and accumulate at mining site. A quantity of residual waste known as “Tailings” (Lottermoser, 2003). These tailings and waste rocks are very large proportion of the original ores. The mining process can be seen in figure 2.1.

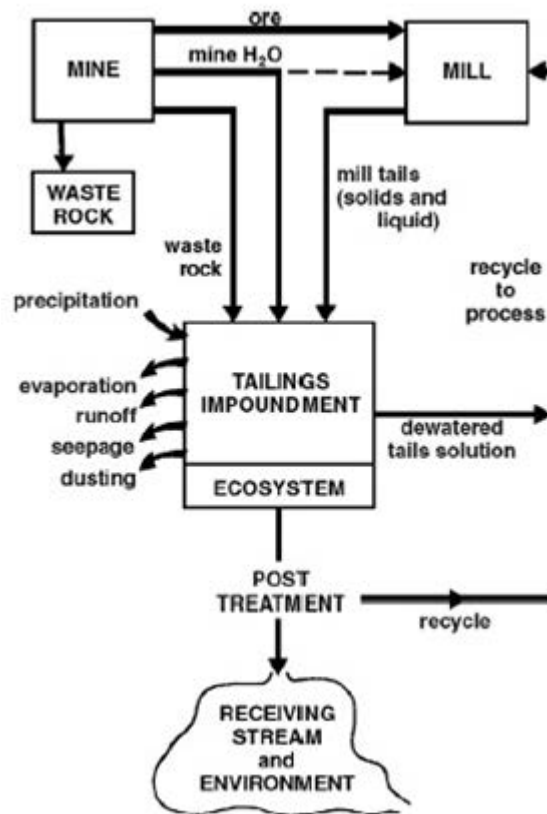


Figure 2.1 Mine/mill tailings environment simplistic system model (Ritcey, 2005)

2.2 Bioavailability

Most chemical immobilization treatments researches have focused on reducing bioavailability, solubility, or extractability. Because of their results may show reduced solubility and potential decreased metal transport. On the other hand, few studies have measured the metal transport reduction from chemical amendments to contaminated soil (Sparks, 2001). Basically, metals in soil are existed as a variety of chemical species in a dynamic equilibrium governed by soil physical, chemical and biological properties (Lasat, 2006). According to several studies (McGowen, 2000; Gobran et al., 2001; Nigam and Srivastava, 2003; Žemberyová et al., 2006), there are, shown the metal form in soil, much greater importance than the total concentration with regard to bioavailability of metals. For example, Gobran et al., (2001) found that the elements behaviors in the environment cannot predict on the basis of their total

concentration because in the fact that not all of the metal may be labile or available for uptaking.

Sequential extractions were used in almost studies to predict the persistence and potential mobility and bioavailability of trace metals. In the first cases, the amounts of trace metals in extractant by a given chemical reagents are supposed to correlate with the amounts accumulated in plants. In the second case, sequential extractions are mainly assumed to provide the localization of trace elements in soils; the reactivity of trace metals in soils are the result from the possible mobility and availability, which depends on their localization in different soil components, which is usually called speciation. McGowen (2000) studied agriculture limestone, mineral rock phosphate, and diammonium phosphate chemical immobilization treatments to reduce Cd, Pb, and Zn solubility and transport in a smelter-contaminated soil. Reducing total Cd, Pb, and Zn elution were found from the contaminated soil. Limestone is effective for reducing Cd and Pb (45% and 54.8% reduction over the untreated check, respectively).

2.3 Sequential extraction

Chemical extraction methods are more sensitive compared to physical methods of speciation. Sequential extraction methods consist of using successively different chemical reagents for trace element extraction from given soil compartments (Belzile et al., 1989; Nirel and Morel, 1990). Many chemical extractions have been developed to determine element behavior in order to fractionate metals. There are single and sequential extractions; single extraction uses only one solvent to extract sample directly. The extractant commonly uses H₂O, KNO₃, EDTA, DPTA and HNO₃. Each extractant is targeted on a single form of each metal. For example, H₂O for soluble material, HNO₃ for each metal residue, and CH₃COOH for bounding particulate material. (Iwegbue et al., 2007)

In this study, the three-step BCR sequential extraction procedure will be used according to the procedure recommended by the Standards, Measurements

and Testing programme of the European Union (SM&T) which are summarized in Table 2.3.1

Mobility and bioavailability of the metals decrease approximately in the order of the extraction sequence (Dudka et al., 1996). Each successive form represents less availability (Iwegbue et al., 2007).

Table 2.1 The three-step BCR sequential extraction scheme (modified from Akkajit, 2008; Pérez and Valiente, 2005; Yang, 2009)

Fraction label	Reagent	Metal species/association
1.Exchangeable (BCR1 or F1)	0.11 mol/L CH ₃ COOH (acetic acid), 16h shaking (22±5 °C)	Bound to colloidal/particulate material or soil surface by relatively weak electrostatic interactions, susceptible to change of pH, released by changes in ionic composition and affected by production or consumption of protons
2.Reducible (BCR2 or F2)	0.5 mol/L HONH ₂ · HCL hydroxylamine hydrochloride at pH1.5, 16h shaking (22±5 °C)	Bound to iron and manganese oxides, instability under anoxic conditions and dissolution of metal-oxide phases under controlled Eh and pH conditions
3.Oxidisable (BCR3 or F3)	8.8 mol/L H ₂ O ₂ (85±2 °C) then 1.0 mol/L CH ₃ COONH ₄ (ammonium acetate) , 16h shaking (22±5 °C)	Bound to organic matter and sulfides various forms. The degradation of organic matter under oxidizing conditions is responsible for trace elements releasing.
4. Residual ^a (F4)	Aqua regia ^a	Concentrated acid digests any chemical bond in substances and all elements be in form of small molecules or solutions

^aThis step is not an official step.

2.4 Soil parameters

2.4.1 Soil pH

Soil pH is affected by the changes in redox potential which occur in soils that become watery. Reducing conditions particularly cause a pH increase, and oxidation leads to a decrease. In general, heavy metal cations are most mobile under acid conditions and increasing the pH by liming usually reduces their bioavailability (Alloway, 1995).

2.4.2 Organic matter

The main organic matter in soils comes from biological decay. The final product of this degradation are humic substances, organic of low-molecular and high-molecular weights, carbohydrates, protein, peptides, amino acids, lipids, waxes, polycyclic hydrocarbons, and lignin fragments. Furthermore, the excretion products of roots, composed a wide variety of simple organic acids, are present in soils (Essington, 2003).

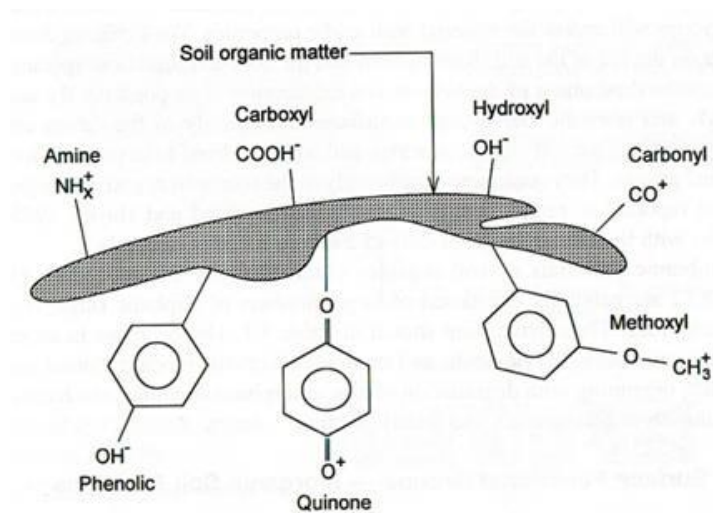


Figure 2.2 Sketch of a soil organic matter (SOM) showing the various kinds of functional group associated with such soil material (Yong, 2002).

The basic structure of all soil organics is formed by carbon bonds that are salicylic or aromatic rings, respectively. See Figure 3, carbon and nitrogen are combined with oxygen and/or hydrogen to form the various types of surface functional groups that properties of organic molecules and their reactions with other eater system. The main functional groups are hydroxyls, carboxyls, phenolic, and amines. They can both protonate or deprotonate depending on the aqueous environment pH, i.e., they will develop positive or negative charges depending on the soil pH. The carboxyl group is the major contributor to the acidic properties of the soil organics (Yong, 2002).

The organic component of soil constituents has a high affinity for metal cations because of the presence of ligands or groups that can chelate metals (Harter and Naidu, 1995). When pH was increased, the carboxyl, phenolic, alcoholic and carbonyl functional groups in soil organic matter dissociate, thereby the affinity of ligand ions for metal cations were increased. The affinity general order for metal cations complexed by organic matter follows: $\text{Cu}^{2+} > \text{Cd}^{2+} > \text{Fe}^{2+} > \text{Pb}^{2+} > \text{Ni}^{2+} > \text{Co}^{2+} > \text{Mn}^{2+} > \text{Zn}^{2+}$ (Adriano, 2001)

Humic substances can serve as carrier of toxic metals, forming complexes that are stable and enhance transport of toxic metal in waters by reducing the toxicity of the heavy metals to the microbes. Especially, inorganic anions such as phosphate and cyanide can be removed from water as well by mixing of ligand complexation (Sparks, 1995).

2.5 The presence of heavy metals

An environment impact of heavy metal in natural has been mainly caused by humans. Man-made sources pollution can easily create local conditions of elevated metal presence which could lead to environmental effects. Although many metallic elements play an essential role in the function of living organism, they constitute a nutritional requirement and fulfill a physiological role, overabundance of the essential trace elements and particularly their substitution by nonessential ones

can cause toxicity symptoms or death. (Volesky, 1990) Heavy metals are important in many ways, for example, using for improving technology in industries of advance countries. Some elements are trace elements and essential for plants and animals. There are many significant as pollutants which effect on ecosystems throughout the world. Heavy metals in soils have gradually increasing in recent years because of scientific and technological growths, including the increasing industrial demands for metals in order to locate or reserves of ore minerals (Alloway, 1995).

Soil is the main component of terrestrial ecosystems. There are many elements in soil, which can be divided into two main group macronutrient and micronutrient (or trace elements). Micronutrients are important in plant nutrition similar to macronutrient but trace elements occur in plants and soils in much smaller concentrations. Trace elements can occur in four main forms in soil: (1) primary and secondary minerals. (2) Adsorbed to mineral and organic matter surfaces. (3) Organic and microbial biomass, and (4) solution. Depending on micronutrients, some forms are more important than others in supplying or buffering plant's available micronutrient in the soil solution. (McLaren, 1997)

2.6 Seven interested metals

2.6.1 Cobalt (Co)

Cobalt is an important element in industrial civilization, and also to maintenance of life within. Co is much less abundant used for the manufacture of special steels, and has been used for centuries in the manufacture of blue pigments and glass. Co has essential roles in living organisms and animals. Environmental pollution problem are relatively insignificant compared with those associated with some other heavy metals. Primary interest in Co as a component of soil lies in its essential roles in ruminant animals and microorganisms. For centuries, farmers in different parts of the world had found some pastures to be unsuitable for sheep and cattle, became weak and emaciated. The symptoms were attributed to low Co Concentrations in the herbage, and top dressing of pastures with Co salts and oral

administration of Co were used to alleviate the condition. Uptake of Co by plants is a function of the concentration of Co in the soil solution and on the exchange sites of cations exchange complex. The reagents used include several which alter significantly one of the two soil properties that have been shown to have the greatest influence on Co availability. Some evidences were found that the uptakes of Co increases as the soil pH decreases. (Spark, 2001)

2.6.2 Copper

Copper is one of the essential elements for organisms. Sometime extractable Cu is called “available Cu”. An abundance of Cu in lithosphere is considered to be 70 mg/kg. Cu is associated with soil organic matter, oxides of Fe and Mn, soil silicate clays and other minerals (Spark, 2001). Chelation and complexing are the key reactions governing Cu behavior in most soils (Kabata-Pendias and Pendias, 2001).

The mainly common available Cu in the surface environment is founded to be the +2 cations valence electron which is very tightly on inorganic and organic exchange sites depending on the surface charge carried by the absorbents (Spark, 2001). There are many reviews of Cu reaction with inorganic and organic components of soils. (Everett et al, 1967; Chow, 1970 and Spark, 2001) The bioavailability of soluble forms of Cu depends most probably on both the molecular weight of Cu complexes and on the amount present. The concentrations of Cu in soil solutions are basically controlled by both the reaction of Cu with active groups at the surface of the solid phase and by reactions of Cu with specific substances (Kabata-Pendias and Pendias, 2001).

2.6.3 Chromium (Cr)

Chromium (Cr) has been used in alloy steels and chrome plating. The most stable common forms are Cr(III) and Cr(VI). Cr(VI) is an anion, more extracted from soil and sediment particles and is considered the more toxic form. Chromate is

in pH-dependent equilibrium with other form of Cr(VI) such as HCrO_4^- and dichromate ($\text{Cr}_2\text{O}_7^{2-}$), with CrO_4^{2-} the predominant form at $\text{pH} > 6$. On the other hand, Cr(III) is much less mobile and adsorbs to particulates more strongly. The solubility of Cr(III) decreases above pH 4, and above pH 5.5 complete precipitation occurs. However, complexing Cr(III) with soluble organic acids such as citric acid, DPTA, fulvic acids and soil extracts of water-soluble organic matter) maintain Cr(III) in solution above the pH at which uncomplexed Cr precipitates and it therefore a means of enhancing its mobility. Concentration of Cr in growing plankton mine spoil and various types of Cr waste are mainly in the range 10-190 mg/kg, but toxic concentrations may accumulate in plants growing on chromate waste in which the more soluble Cr(VI) form predominates. (Sparks, 2001)

2.6.4 Manganese (Mn)

In Lithosphere, Manganese (Mn) is one of the most abundant trace elements. Mn in form of various oxide and hydroxide species and chelates are found in many components which some are simple and complex ions in solution, and also several oxides of variable composition. Mn exists in multiple oxidation states: Mn(II), Mn(III) and Mn(IV) but the Mn(II) is the most frequent found in the rock-forming silicate minerals. All Mn compounds are very important soil constituents because this element is essential in plant nutrition and controls the behavior of several other micronutrients. It was found that Mn uptake is metabolically controlled which was similar to that of divalent cations species such as Mg^{2+} and Ca^{2+} and known to replace the sites of some divalent cations (Fe^{2+} , Mg^{2+}) in silicates and oxides (Kabata-Pendias and Pendias, 2001).

The Mn compounds are known for their rapid oxidation and reduction under variable soil environment and thus oxidizing condition may reduce the availability of Mn and associated micronutrients, whereas reducing conditions may lead to the ready availability of these elements even up to toxic range. The complex mineralogical and chemical behavior of Mn results in the formation of the large

number of oxides and hydroxides which give a continuous series of composition of stable and meta-stable arrangements of atoms. Colloidal Mn oxides reveal a great affinity for adsorption of cationic and anionic forms elements as well as inorganic and organic substances (Kabata-Pendias and Pendias, 2001).

2.6.5 Nickel (Ni)

Nickel (Ni) can occur in a number of oxidation states, but only Ni(II) is stable over the wide range of pH and redox conditions found in the soil environment. Ni can be replaced essential metals in metallo-enzymes and cause disruption of metabolic pathways. The average concentration of Ni is 40 mg/Kg which varies much between soil types. The content of Ni depends on nature of material. The soil chemistry of Ni is much simpler and is based on the divalent metal ion (Ni²⁺). The solubility of Hydroxides of Cr(Cr(III)) and Ni, both are siderophilic elements. At difference of pH values give some indication of the relative mobility of these entities in soils. Both become increasingly soluble at lower pH values, but clearly Ni is the more soluble. (Sparks, 2001)

2.6.6 Lead (Pb)

Lead (Pb) is not essential for plants and exists in three oxidation states: Pb, Pb(II), and Pb(IV). Lead exists in all soils and all crops which tend to accumulate in the soil surface. The natural Pb content of soils is reported to be the least mobile among the heavy metals and occurs mainly as Pb²⁺ (Kabata-Pendias and Pendias, 2001). The major portion is usually solid or adsorbed onto soil particles as long as reducing conditions are maintained. Plants tolerance to soil lead is very high because it is easily adsorbed, once incorporated into the soil; lead tends to have a low mobility, resulting in a long residence time (Haroun et al., 2006).

The great variation of Pb contents of plants is influenced by environmental factors which are known to promote both Pb uptake by roots and Pb translocation into plant top (Kabata-Pendias and Pendias, 2001). The concentration of lead in plants depends on their total and available concentrations in soil, soil

properties, and plant species, age, and cultivar, plant parts, plant species and the type of amendments (Chlopecka and Adriano, 1997). A high soil pH may precipitate Pb as hydroxide, phosphate, or carbonate, as well as promote the formation of Pb-organic complexes which are rather stable. Increasing acidity increases the Pb solubility (Kabata-Pendias and Pendias, 2001). Lead is tightly bound under strongly reducing conditions by sulfide mineral precipitation and complexation with insoluble organic matter, and is very effectively immobilized by precipitated iron oxide minerals under well-oxidized conditions.

2.6.7. Zinc (Zn)

Zinc is essential for plants and animals and is considered to be readily soluble relative to the other heavy metals in soils (Kabata-Pendias and Pendias, 2001). Zinc is unique among the quartet of metals (Cu, Fe, Mn, and Zn) as zinc does not exhibit multiple valences and is not subject to oxidation-reduction in the soil plant system (Miller and Gardiner, 2001). Zn is relatively active in biochemical processes and is known to be involved in several biological and chemical interactions with several elements (Kabata-Pendias and Pendias, 2001).

A zinc tissue concentration of less than 15 mg/kg (dry weight) leaves a plant deficient, whereas a concentration over 400 mg/kg (dry weight) is potentially phytotoxic. Toxicities of zinc are seldom observed until plant tissue levels in excess of 1000 mg/kg are reached (Haroun et al., 2006). The most common and mobile Zn in soil is believed to be in forms of free and complexed ions in soil solutions (Kabata-Pendias and Pendias, 2001). Zinc may form complexes, for example $Zn(OH)_2$, $Zn(OH)_3$, and $Zn(OH)_4$ with OH^- depending on the pH and metal concentration (Ören and Kaya, 2006). As a result of high pH value, zinc hydroxyl species may participate in the adsorption and precipitation. Zinc forms complexes with inorganic and organic ligands, which will affect its adsorption reactions with the soil surface. The adsorption of Zn^{2+} can be reduced at lower pH by competing cations and these results in easy mobilization and leaching of Zn from light acid soils.

CHAPTER III

METHODOLOGY

3.1 Introduction

In this research, soil tailings samples were collected in late April 2010 for conducting further experiments to investigate heavy metal fractionation. Ten tailing samples have been taken from the one-meter depth below ground surface. The experimental design in this study consists of two main parts as shown in the experiment diagram (Figure 3.1). All the apparatus used is given in Appendix A.

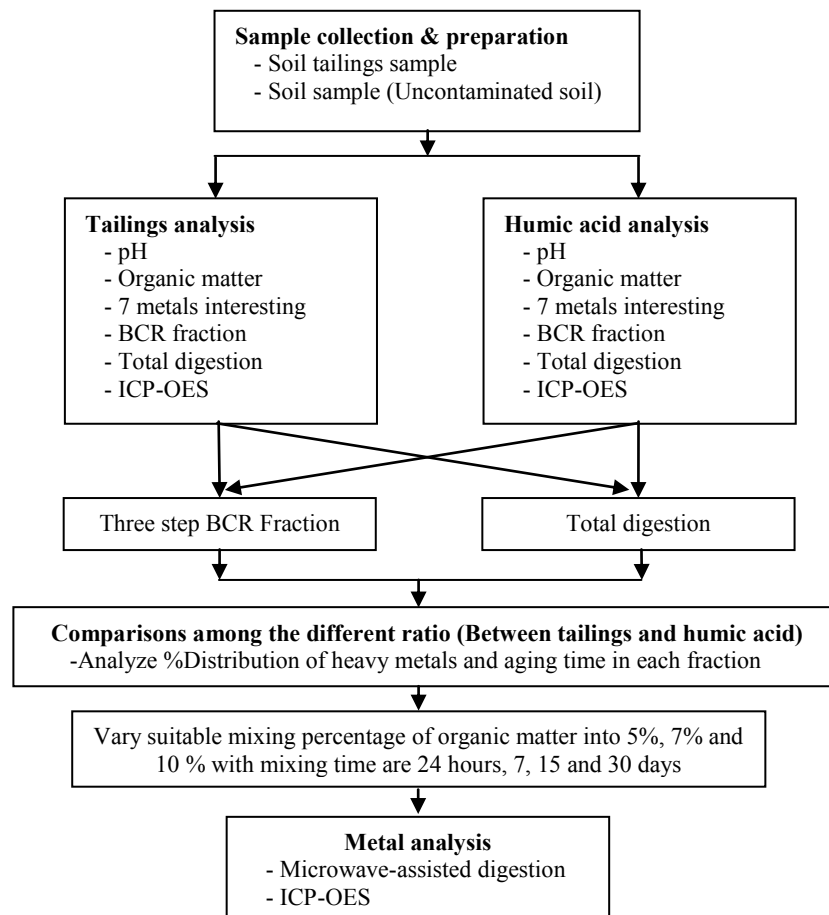


Figure 3.1: Experimental design in this study

3.2 Study Sites

The Sampling sites located in TSF area, Akara gold mine, Pichit provinces. All details of sampling sites are given in Appendix B.

3.3 Sample collection and preparation

3.3.1 Soil sample collection and preparation

All samples were immediately stored and sealed in polypropylene bags and kept at 4 °C before taken to the laboratory for further processing and analysis. At the laboratory, samples were air dried until a constant weight is achieved. Then dried sample were crushed into fine powder using an agate mortar before passing through a 200-mesh sieve. They were carefully screened and put in polypropylene zip-lock bag until analysis. The particles less than 200 mesh ($<74\mu\text{m}$) in soil samples, which are representative of real soil, was used for analysis in this study and stored in polyethylene bottles in a desiccators at room temperature prior analysis (Tokalioglu, 2003). Precautions were taken to avoid contamination during sampling, drying, grinding, sieving, and storing.

3.3.2 Preparing soil samples into soil amendment

Soil amendment samples were mixed between the organic matter percentage of humic acid and soil tailings into 5%, 7% and 10% (weight by weight) and separate into three conditions; Samples were inoculated at 24 hours, 7, 15 and 30 days respectively. Twelve conditions (including control sample; only used tailings samples, no adding Humic acid) of soils inoculated was investigated fractionation of heavy metals in each sample using three-step BCR sequential extraction and Microwave digestion (for their residues), and then analyzing with Inductively Couple plasma-Optical Emission Spectrometer (ICP-OES).

3.4 Analysis parameter

3.4.1 Soil chemical properties

pH: pH was determined by using a pH meter.

3.4.2 Organic matter

Dried soil samples was digested and followed by the Walkley-Black method as below;

0.5 g soil sample was added into a 500 mL conical flask, and 10 mL of 1N K₂Cr₂O₇ was then added through a pipette. The soil and dichromate were slightly swirling mixed in the flask; meanwhile 20 mL of concentrated H₂SO₄ was also added. The slightly swirling in the flask was performed in order to allow soil and reagents having a well mixing.

The content of the flask was held for 30 minutes, followed by dilution with 200 mL of water, 10 mL of 85% H₃PO₄, 0.2 g of NaF, and 10 drops of diphenylamine indicator; the solution is a violet blue. Then, titrating with 0.5 M ferrous sulfate solution, the solution becomes green at the end point. The organic carbon and organic matter in soil can be calculated by using the following equations.

(1) Percent Organic Carbon

$$\% \text{ Organic C} = \frac{(B-S) \times M \text{ of Fe}^{2+} \times 0.336}{\text{g of soil}} \quad \dots[1]$$

Where;

B = mL of Fe²⁺ solution used to titrate blank

S = mL of Fe²⁺ solution used to titrate sample

(2) Percent Organic Matter

$$\% \text{ Organic Matter} = 1.72 \times \% \text{ Organic C} \quad \dots[2]$$

Where;

% Organic C = value is calculated by using the Equation [2]

3.4.3 Calculating soil organic amendment (%SOM)

Percentage of soil organic amendment used percent organic matter in above equations to get the final proportion of mixing soil as following:

$$(a \times 1) + (X \times 1) = b (1+X)$$

Where;

a = %Organic matter from 3.4.2

b = final %SOM

3.5 Metal analysis

The concentrations of seven metals in the samples were analyzed as available fraction and total concentration by using a three-step BCR sequential extraction method and microwave assisted acid digestion, respectively. The plastic containers and glassware used in the experiment was carefully washed and soaked in 5% HNO₃ solution overnight followed by repeating washing steps with deionized water. (18.2 MΩ)

3.5.1 BCR sequential extraction

The sequential extraction procedure that will be applied in this study is composed of three steps based upon the Standards, Measurements and testing programme of the European Union (SM&T). Three-step BCR sequential extraction scheme has been widely used in different types of sediments, contaminated soil, industrially-contaminated made-up ground, sewage sludge and fly ashes. For quantifying the fraction of metal characterized by the highest mobility and availability, a high enough total metal concentration was strongly recommended for BCR procedure that applied to sediments. There is a comparison between three-step BCR and modified 3step BCR scheme. The difference concentration of NH₂OH.HCL in the second step proved to be the most relevant, especially for Cr, Cu, and Pb extraction which showed a dramatic decrease in both extractability and reproducibility as pH increased. The rest of studied factors did not show significant effect upon reproducibility. The use of NH₂OH.HCL 0.5 mol/L adjusted to pH 1.5 by using a

fixed volume of diluted HNO₃ to the extractant solution and the centrifugation speed was increased from 1500 to 3000g for every step. The combination increases the concentration of NH₂OH.HCL from 0.1 to 0.5 mol/L in the modified scheme with a pH lowered to 1.5 also providing a better release of metals bound to hydrous oxides of iron whereas the original reagent largely attacked only the hydrous oxides of manganese. (Quevauviller, 2002) For analyzing varieties of heavy metal in preliminary test, there are many elements to determine; therefore, the three step modified was used in the preliminary step and throughout the study. Each extraction step details are described in the following paragraphs.

BCR1: Exchangeable /Acid soluble fraction

An acetic acid solution (20 ml of 0.11 Mol/L solutions) was added into the polypropylene centrifuge tubes (soil: solution ratio is 1:40) and shaken for 16 hours at room temperature (22 ± 5 °C). No delay should be occurred between the addition of the extractant solution and the beginning of the shaking. The extract was separated from the solid residue by centrifugation for 15 minutes at 3000 rpm, and the supernatant liquid were decanted into a polyethylene container and stored in a refrigerator at 4 °C until analysis. The residues were washed with 20 mL deionized water, shaken for 5 minutes on an end-over-end shaker and centrifuged for 15 minutes at 3000 rpm. After decanting the supernatant, the residues were ready for the next step (BCR1).

BCR 2: Reducible fraction

To the residues from Step 1 (BCR1), 20 mL of hydroxylamine hydrochloride (0.1Mol/L) were added (soil: solution ratio is 1:40) and then shaken for 16 hours at room temperature (22 ± 5 °C). No delay should be occurred between the addition of the extractant solution and the beginning of the shaking. The extractants were separated from the solid phase by centrifugation and decantation as described for BCR1. The residues were washed with 10 mL deionized water, shaken for 15 minutes on an end-over-end shaker and centrifuged for 15 minutes at 3000 rpm. The supernatant were decanted and stored at 4 °C until analysis.

BCR 3: Oxidizable fraction

To the residues from Step 2 (BCR2), 8.8 Mol/L hydrogen peroxide were added at a soil solution ratio of 1:10 and then shaken for 1 hour at room temperature (22 ± 5 °C) by end-over-end shaker. The solution will be heated up to 85 °C for 1 hour. The extraction is repeated by using the same procedure. Then, the sample was shaken for 16 hours at 350 rpm at room temperature (22 ± 5 °C). The extract was then separated from the solid phase by centrifugation and decantation as described for BCR1. The supernatant was decanted and stored at 4 °C (Tokalioglu et al., 2003 and Yang et al, 2009). Microwave assisted digestion and analyzed element concentration with Inductively Coupled Plasma-Optical Emission Spectrometers (ICP-OES) were used for determination the metal concentration of the residue from BCR3 as described in 3.5.2.

3.5.2 Total concentration

For estimation of heavy metals concentration from BCR final residue in soil, samples was directly digested by using the microwave assisted digestion procedure according to the aqua regia method.

Dried soil 0.5 g in PTFE vessels 14.5 mL of aqua regia solution was added (modified from microwave digestion application note manual). Aqua regia solution was the mixed acid of concentrated hydrochloric acid and concentrated nitric acid at a ratio 3:1. The digestion by aqua regia was performed at high pressure and temperature. The recommended temperature followed in two steps: the first step was a raising temperature, in this step the temperature increased from room temperature to 180 °C within 10 minutes; and the second step was the holding temperature which means that the temperature holds at 180 °C throughout digestion for 20 minutes. Then, samples was cooled down and filtered by using a Whatman No.41 filter paper. The volume was adjusted to 50 mL with deionized water in volumetric flask. Total heavy metal concentration was measured by Inductively Coupled Plasma-Optical Emission Spectrometers (ICP-OES).

To validate the method, the accuracy of the total digestion procedures for determining metals in the extracts was compared to the results of the Certified Reference Material, CRM 025-050 (RTC).

3.6 Soil amendment analysis

Before preparation of soil amendment samples, humic acid; an analytical grade, was weighted and mixed with dried tailings samples. Preparation and analysis of humic acid used the same method as soil analysis.

3.7 Quality control

Certified reference material was also analyzed following the same method as the sample in order to provide an indication of the extraction efficiency of each method. The analytical procedure accuracy for total and sequential extraction was checked using a soil certified reference material. CRM 025-050 (RTC) was moderately contaminated soil available from the Western United States (Peter Matušík et al., 2005). All samples and reference materials were carried out in triplicate for each experiment.

3.8 Data analysis

Statistical studies were applied for determination the correlation analysis by Statistical software SPSS packages program. Seven metals were interested in different soil amendments aging ratio and percent of leachate in each fraction step were analyzed and calculated based on the correlation matrix.

CHAPTER IV

RESULTS AND DISCUSSION

4.1 The Physicochemical characteristics of soil and humic acid

The physicochemical characteristics of 30 samples from ten sampling spots (Fig. 4.1 to 4.3) are summarized in table 4.1. There are more details of raw data in table C-1 and C-2 in the Appendix C. Soil samples show physicochemical properties: pH ranges from 8.00 to 8.45 and organic matter content (%OM) from 1.04 to 1.50. In the experiment, Humic acid was bought from Sigma-Aldrich Corporation in Technical grade, pH ranges of 3.0, after rechecked at lab pH ranges of 1.97 to 2.07. The organic matter content ranges of humic acid are 70.05 to 78.33. Cations of heavy metal mostly mobile under acid condition and pH can be increased by liming usually reduces their bioavailability (Alloway, 1990)

Table 4.1 Physicochemical parameter of Soil tailings and Humic acid

Statistic value	Soil Tailings		Humic acid	
	pH	OM (%)	pH	OM (%)
Min	8.00	1.04	1.97	70.05
Max	8.45	1.50	2.07	78.33
Mean±SD	8.25±0.19	1.27±0.18	2.02±0.05	74.59±3.66
Median	8.25	1.23	2.03	75.39



Figure 4.1 Ten locations in Tailing Storage Facilities, TSF (small rectangular pictures)



Figure 4.2 The TSF in Akara gold mine.



Figure 4.3 How to sample soil tailings samples from TSF area in each punch.

For soil organic matter contents, almost soil tailings ranges are very low (1.04- 1.50%) which indicate that soil tailings in this area does not fertile because most agriculture soil is between 2-10% (Alloway, 1990).

4.2 Total metal concentrations in soil tailings

All samples were air dried, passed through a 200-mesh sieve and used three steps BCR sequential extraction from ten locations. All residues from oxidizable fraction or BCR3 were digested by using microwave digestion techniques, EPA 3052 method and aqua regia, in order to determine the total metal concentrations. The maximum, minimum, mean and median total metal concentrations (mg/kg soil), and standard deviations (%) of seven interested elements (Co, Cr, Cu, Mn, Ni, Pb and Zn) obtained from determination of 30 samples are reported in Table 4.2 and the comprehensive analytical results were shown in Appendix D-1.

All above data were shown in Table 4.2. and Fig 4.5 to 4.11. For Fig.4.5 to 4.11 were shown in bar graph, each bar include 4 fractions; BCR1, BCR2, BCR3 and residual fraction into percentage of fraction which calculating from average element concentration of raw data. X-axis was sampling locations and Y-axis was 100%fraction. Results found 70-80% of extractable Mn and 50-60% of extractable Pb in BCR1 and BCR2 fraction from whole fraction. The other was found in residual fraction (Pb Ni and Zn (50-60% of all fractions), Cr and Cu (70-80% of all fractions)).

Each fraction was considered and there are some researches published that almost first and second fractions, exchangeable and reducible fractions, were dissolved easily into environment (Bacon and Davidson, 2008, Akkajit and Tongcumpou, 2010). Metal concentrations results from BCR1 and BCR2 were compared with Soil quality standards for habitat and agriculture of Pollution control department in Thailand of Cr, Mn, Ni, and Pb are not exceed 300, 1800, 1,600 and 400 mg/kg, respectively (PCD, 2008). On the other hand, the metal concentrations were determined from BCR1 and BCR2 mean elements can be leached into

environment. Chotpantararat et al., (2008) and Changul et al (2008) found that concentration of heavy metal (Mn and Pb) in gold mine area are exceed than water quality standards for Industrial effluent Standards of Pollution control department in Thailand, which of Cu, Mn, Ni, and Pb are not more than 2.0, 5.0, 1.0, and 0.2 mg/L (PCD, 2004), reported in ground water concentration value exceeded the water quality standards for Industrial effluent Standards of Pollution control department in Thailand (See Appendix B and Fig. 4.4 to 4.10).

Table 4.2 Descriptive statistics of element concentrations (mg/kg) in 120 soil tailings samples by three-step BCR sequential extraction and total digestion

BCR1	Metal concentrations in samples (mg/kg)						
	Co	Cr	Cu	Mn	Ni	Pb	Zn
Max	2.86	0.63	7.95	629.86	3.01	3.97	18.34
Min	1.54	0.41	5.13	461.98	1.15	0.90	9.50
Mean	2.32	0.50	6.75	577.37	1.63	1.85	13.28
±SD	±0.39	±0.06	±0.65	±47.42	±0.34	± 0.90	± 2.35
BCR2	Co	Cr	Cu	Mn	Ni	Pb	Zn
Max	16.63	18.54	12.46	793.37	8.70	34.94	96.22
Min	11.80	7.67	5.90	436.58	2.97	19.86	7.45
Mean	13.85	13.90	10.10	677.57	4.46	28.63	13.80
±SD	± 1.17	± 2.00	± 1.53	± 88.44	± 0.96	± 3.72	± 15.96
BCR3	Co	Cr	Cu	Mn	Ni	Pb	Zn
Max	12.05	8.11	17.81	36.83	8.33	14.49	146.59
Min	0.40	0.87	2.64	0.07	0.59	2.10	0.12
Mean	1.82	2.45	5.08	17.07	2.08	6.02	9.14
±SD	± 2.82	± 1.63	± 3.14	± 12.16	± 1.43	± 3.05	± 27.54
RES	Co	Cr	Cu	Mn	Ni	Pb	Zn
Max	11.01	110.03	68.59	501.44	13.62	73.36	152.09
Min	6.70	59.92	37.23	335.32	7.26	9.51	55.07
Mean	8.79	88.86	54.35	429.47	9.53	21.38	82.70
±SD	± 1.25	± 12.23	± 7.62	± 47.70	± 1.59	± 15.06	± 21.31

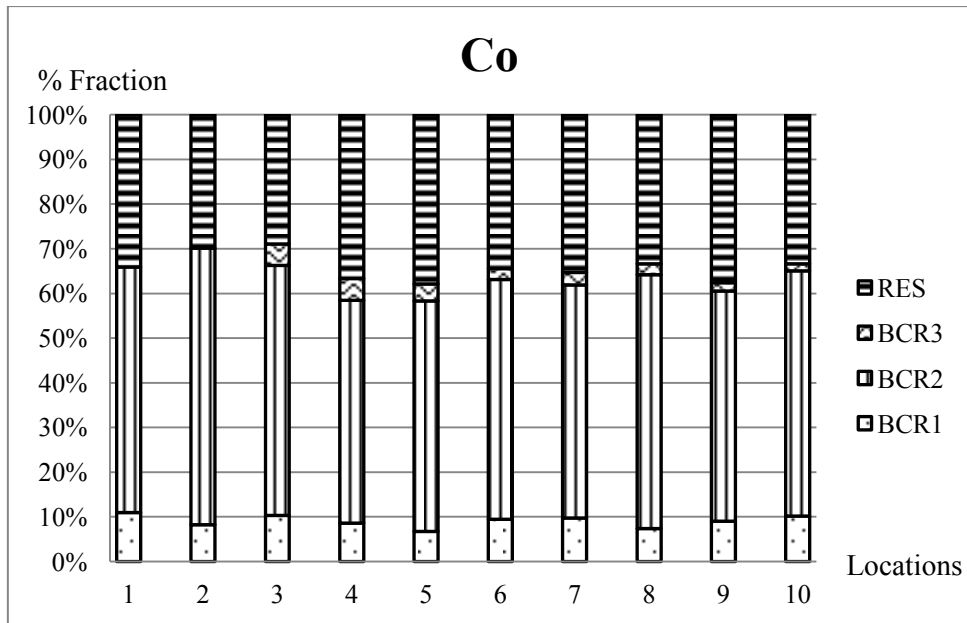


Figure 4.4 Average Co concentration of all fraction from ten locations

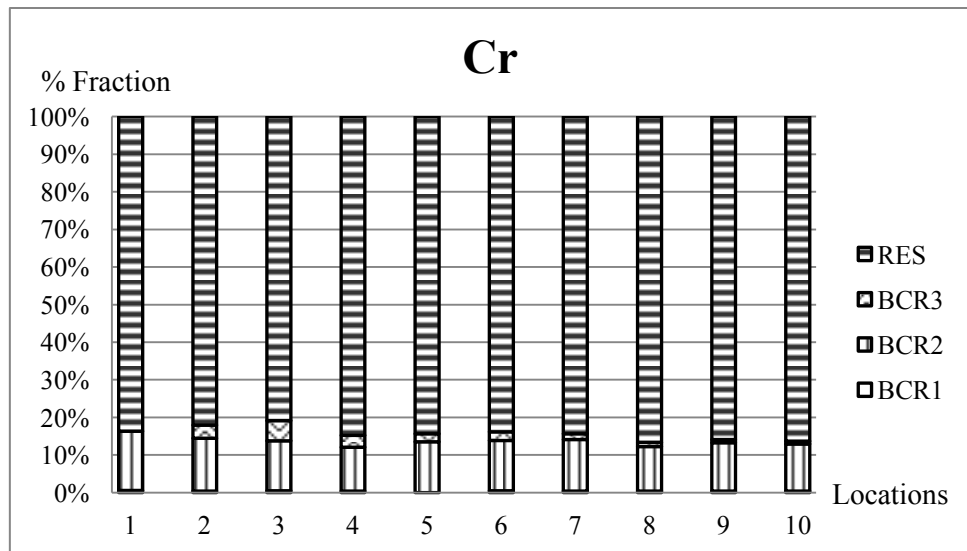


Figure 4.5 Average Cr concentration of all fraction from ten locations

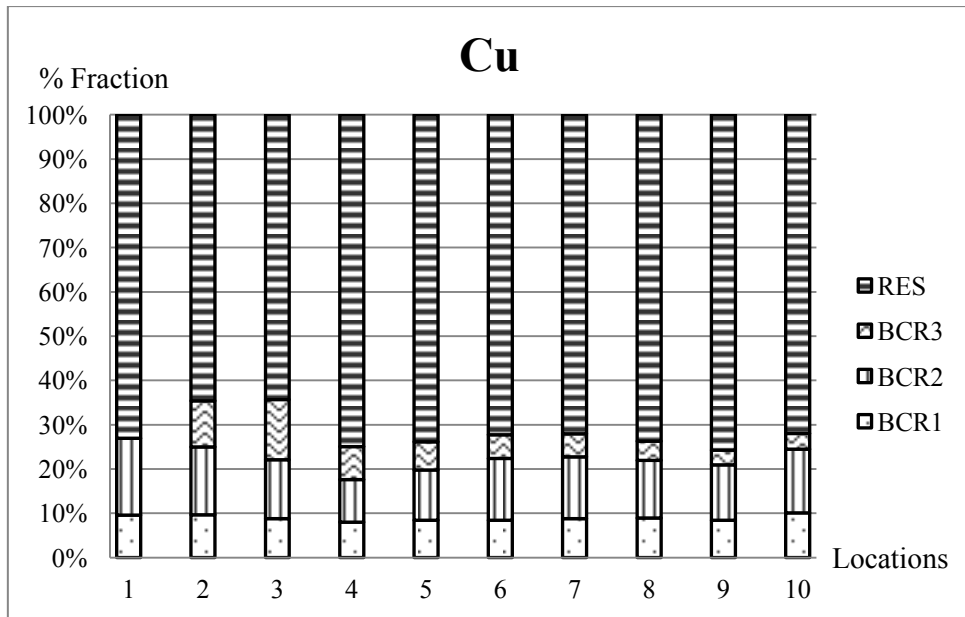


Figure 4.6 Average Cu concentration of all fraction from ten locations

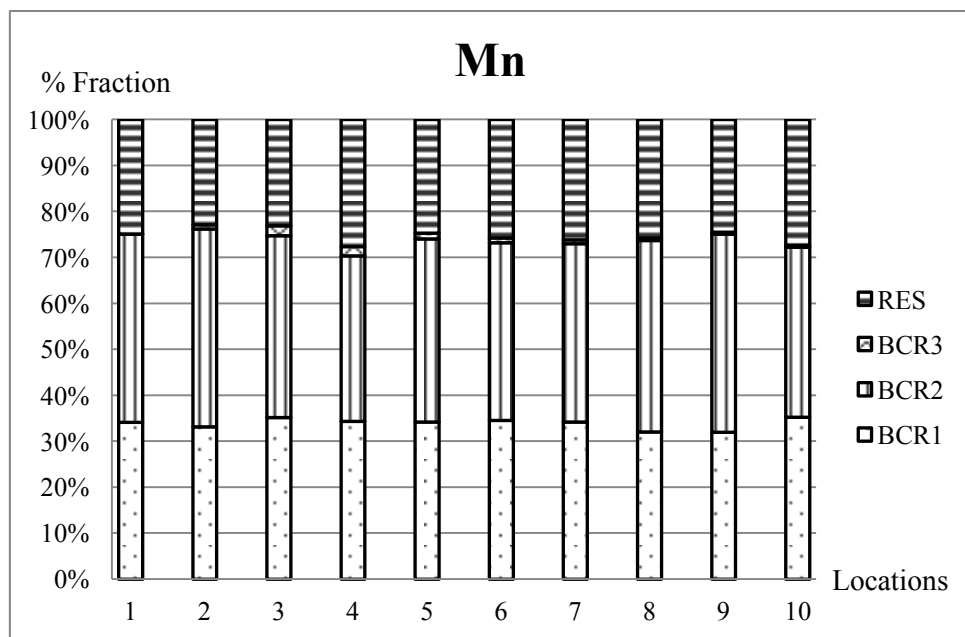


Figure 4.7 Average Mn concentration of all fraction from ten locations

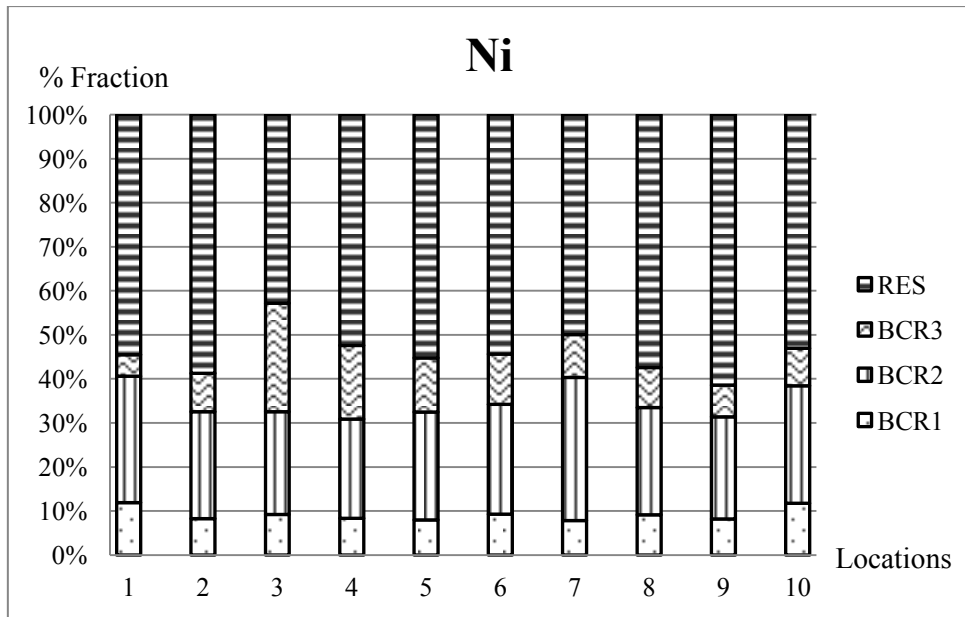


Figure 4.8 Average Ni concentrations of all fraction from ten locations

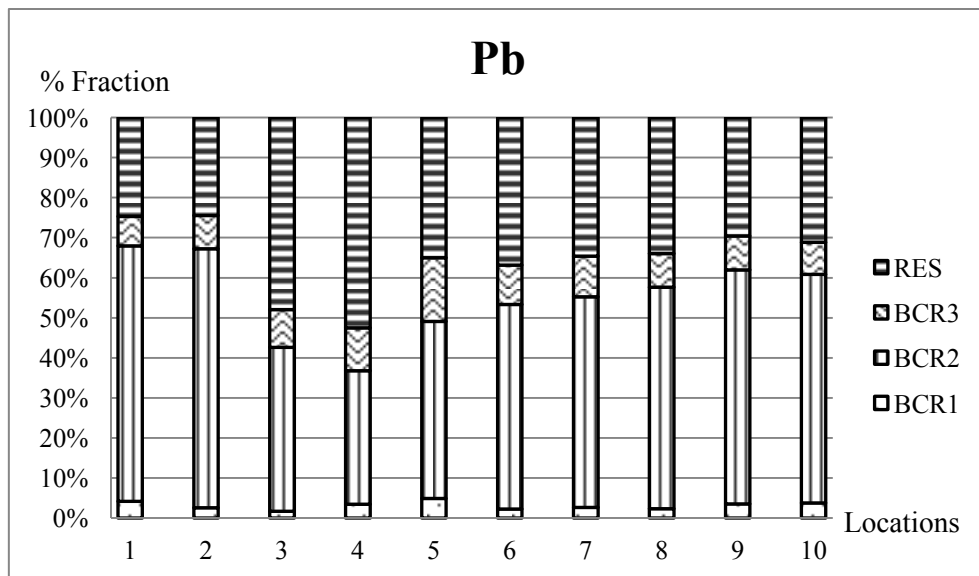


Figure 4.9 Average Pb concentration of all fraction from ten locations

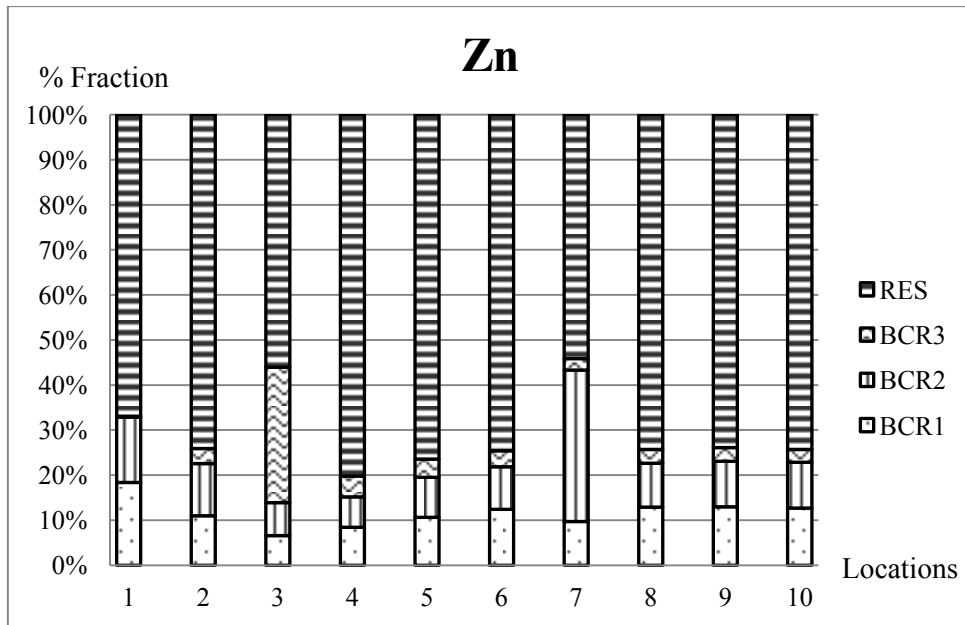


Figure 4.10 Average Zn concentration of all fraction from ten locations

From those average data of seven metal elements from ten sampling spots in TSF. Data was determined in percentage of BCR1 and BCR2 by using formula;

$$\%BCR1 = \frac{BCR1 \times 100}{(BCR1 + BCR2 + BCR3 + Res)}$$

$$\%BCR2 = \frac{BCR2 \times 100}{(BCR1 + BCR2 + BCR3 + Res)}$$

There were shown summary data in Table 4.3 which appeared from maximum to minimum percentage; for percentage of BCR1: Mn (32.39) > Co (8.88) > Ni (8.20) > Cu (8.11) > Zn (6.80) > Pb (3.85) > Cr (0.45), for percentage of BCR2; Co (51.90) > Mn (40.82) > Pb (34.91) > Ni (26.40) > Zn (22.62) > Cr (13.27) > Cu (12.33), and Summary proportion between BCR1 and BCR2: Mn (73.11) > Co (60.78) > Pb (38.76) > Ni (34.60) > Zn (29.42) > Cu (20.43) > Cr (13.72).

Table 4.3 Average percentage proportion of tailing from ten locations in BCR1 and BCR2 from maximum to minimum.

Tailings from ten locations									
Element	Conc.	%(Max > Min)	(Max > Min) Element	Element	Conc.	%(Max > Min)	(Max > Min) Element	% (Max > Min)	
BCR1	BCR1	BCR1	BCR1	BCR2	BCR2	BCR2	BCR2	BCR1+BCR2	
Mn	622.9095	32.29	Mn	Mn	787.4717	40.82	Co	Mn	73.11
Zn	16.1010	6.80	Co	Zn	53.5845	22.62	Mn	Co	60.78
Cu	7.6769	8.11	Ni	Cu	11.6719	12.33	Pb	Pb	38.76
Pb	3.7400	3.85	Cu	Pb	33.9403	34.91	Ni	Ni	34.60
Co	2.7300	8.88	Zn	Co	15.9478	51.90	Zn	Zn	29.42
Ni	2.0848	8.20	Pb	Ni	6.7126	26.40	Cr	Cu	20.43
Cr	0.5547	0.45	Cr	Cr	16.4584	13.27	Cu	Cr	13.72

4.3 Validation of the method

This experiment has a quality control by using natural matrix certified reference material (CRM) standard soil 025-050 which show metal concentration in detail on Appendix Table D-1, and compare between standard deviation averages CRM which measure in experiment as quality control show in Table 4.4. The table shows almost elements of measure values are in range of prediction interval, excluding Pb in range of confidential interval.

Table 4.4 Metal concentrations (mg/kg) of Certified and measured reference material (CRM 025-050)

Elements	Measure values (mg/kg)	Confidential Interval (mg/kg)	Prediction interval (mg/kg)
Co	6.54 ±0.50	3.56 - 4.57	2.03 - 6.10
Cr	497.53 ±14.85	419 - 463	335 - 547
Cu	18.05 ±1.59	7.03 - 8.48	4.21 -11.3
Mn	252.02 ±21.07	167 - 180	141 - 205
Ni	27.12 ±1.17	10.7 - 13.6	4.69 - 19.6
Pb	1,450.16 ±217.11	1,359 -1,536	1,017- 1,878
Zn	76.82 ±2.28	48.4 - 55.1	34.3 - 69.2

4.4 Comparison between aging time and organic amendment ratio

In this study, soil amendment with different ratio of humic acid and soil tailings were carried out for 30 days. Samples were collected after 1-, 7-, 15-, and 30-day-incubation with mixing soils into 5%, 7% and 10% SOM (by calculating results from 4.1). All elements concentration data in triplicate were shown in

Appendix D-2 to D-5 and average concentration of data (Table 4.4) and graphs (Fig. 4.11 to 4.45). All graphs and data are shown in each element and fraction in the following paragraphs by using SPSS statistics program to compare significant differences among average control and other SOM ratio. Statistical analysis was performed, based on these data by using select cases function button to separate and analyze in each incubation day ONE-WAY ANOVA models and LSD (Least Significant Difference), respectively. Table 4.4 was shown fraction by fraction from BCR1 as 1, BCR2 as 2, BCR3 as 3 and residual fraction as 4, and all seven elements concentration were shown in unit of mg/kg soil. For seven elements, results were shown in each element and organized by results of BCR1, BCR2, BCR3, Res (Residual fraction) and all proportion of fraction in each element in 100 percentages. By using this formula to calculate a percentage of decreasing or increasing when compare with average control value:

$$\% = \frac{[\text{Average value at x day}] - [\text{Average value of control}] \times 100}{[\text{Average value of control}]}$$

Table 4.4 Average all elements concentrations (mg/kg soil) in 1, 7, 15 and 30 days in all fraction

BCR	Day	SOM	Co	Cr	Cu	Mn	Ni	Pb	Zn
1	1	Ctrl	2.8469	0.7595	6.6867	596.4959	1.4435	1.4428	8.1935
1		5%	2.6994	0.7309	0.9934	613.4820	2.0592	0.7383	7.3844
1		7%	2.4922	0.5604	0.7560	624.8585	2.5721	0.4452	7.0629
1		10%	2.3854	0.6091	0.5477	618.1676	2.0973	0.4001	6.8140
1	7	Ctrl	2.6419	0.6729	5.9004	572.4345	1.6538	1.5097	7.8153
1		5%	2.7361	0.7418	0.8539	642.5817	2.2016	0.8182	7.2616
1		7%	2.6690	0.5544	0.6211	662.2470	2.2882	0.4773	6.6801
1		10%	2.4731	0.6363	0.5174	647.2612	2.2759	0.2338	6.6023
1	15	Ctrl	3.0691	0.6275	6.3345	528.4903	1.7263	1.1493	8.9761
1		5%	3.0974	0.7751	0.8158	596.3625	2.1973	0.9281	8.1245
1		7%	2.8234	0.5911	0.6229	580.1459	2.6731	0.5334	7.6743
1		10%	2.6510	0.6587	0.5383	555.9758	2.3938	1.0636	7.0005
1	30	Ctrl	2.8096	0.5878	5.0764	638.3723	2.0671	1.2935	8.9209
1		5%	2.9650	0.5933	0.5603	744.6370	2.7530	0.5897	7.9737
1		7%	2.6030	0.4795	0.4603	704.3332	2.8910	0.4601	7.8521
1		10%	2.3543	0.6464	0.4362	700.7912	2.9029	0.3105	6.8439
BCR	Day	SOM	Co	Cr	Cu	Mn	Ni	Pb	Zn
2	1	Ctrl	4.7363	14.8643	11.7532	765.3267	4.3103	21.8454	8.9507
2		5%	4.7323	11.8420	5.0148	737.0984	4.7368	20.2629	9.9156
2		7%	5.0247	11.6000	2.8964	707.2374	5.2423	17.9014	10.9775
2		10%	4.8863	10.3171	2.2006	686.5575	5.2279	16.5019	10.9783
2	7	Ctrl	4.1500	13.8248	11.5626	705.4645	3.8705	21.4014	8.3003
2		5%	4.1597	12.5626	4.5681	646.0223	4.2593	20.3170	9.3958
2		7%	4.5109	11.9767	2.6505	640.5451	4.7344	18.4009	11.3384
2		10%	4.7359	11.4543	2.0405	651.5717	4.9694	17.7160	10.4946
2	15	Ctrl	3.9065	16.2015	10.0466	633.9519	4.2838	20.2410	9.7308
2		5%	4.8843	14.0183	3.6045	612.2350	5.0220	18.6710	11.4458
2		7%	5.8932	13.3647	2.5728	674.5862	5.6365	17.2250	13.5405
2		10%	5.0801	12.6473	1.5969	570.3622	5.4186	16.0520	11.8688
2	30	Ctrl	3.9629	20.8031	12.3216	867.6175	3.2819	18.9490	11.9995
2		5%	4.8403	19.7349	5.8887	825.0901	4.5077	19.9050	16.1485
2		7%	5.9769	17.6945	4.1735	827.3822	4.6226	19.1790	15.3935
2		10%	4.3804	17.3254	2.8662	782.9268	4.6994	16.7510	14.5621

Table 4.4 Average all elements concentrations (mg/kg soil) in 1, 7, 15 and 30 days in all fraction (Cont.)

BCR	Day	SOM	Co	Cr	Cu	Mn	Ni	Pb	Zn
3	1	Ctrl	5.3923	3.1596	4.8292	16.2944	1.3258	3.3980	4.3538
3		5%	7.9683	4.2500	11.4133	26.3916	2.0162	5.2725	4.9215
3		7%	8.1539	5.4866	13.9901	33.7537	2.4531	5.4765	5.6362
3		10%	6.9660	7.0323	13.0124	31.3800	2.2836	6.6674	6.2876
3	7	Ctrl	6.2781	3.0173	6.5415	16.5347	1.4239	2.3548	4.2812
3		5%	6.9469	4.7189	11.6604	25.7213	2.0282	4.3354	5.6128
3		7%	8.4119	6.4568	14.5573	31.3225	2.5299	4.4776	6.3465
3		10%	8.3913	7.1877	14.8735	35.2674	2.7873	5.7970	7.0523
3	15	Ctrl	5.8074	3.7101	6.1702	24.3973	1.7806	2.3548	6.6429
3		5%	7.7224	4.6449	10.7743	48.7897	1.9600	4.3354	9.9758
3		7%	9.3467	5.8045	13.2725	61.3927	1.7485	4.4776	8.6586
3		10%	10.4613	8.9039	13.7481	69.8169	1.9554	5.7970	10.5438
3	30	Ctrl	5.4733	2.6515	5.2376	10.8135	1.7337	2.2516	4.6381
3		5%	6.8096	3.8632	12.3188	16.0173	1.8023	2.9162	5.6041
3		7%	7.1838	5.6404	13.2946	18.7927	1.9275	4.0391	5.8754
3		10%	6.5115	5.7808	13.3571	20.8265	1.8495	3.8589	7.0449
BCR	Day	SOM	Co	Cr	Cu	Mn	Ni	Pb	Zn
4	1	Ctrl	7.6190	86.3081	58.6394	430.2268	9.0224	11.9751	68.8165
4		5%	6.4058	80.7567	53.9556	418.0158	8.3070	13.3171	68.8514
4		7%	5.8986	79.6961	59.9173	386.1582	8.5229	15.7301	89.9839
4		10%	6.6450	82.4337	53.6011	407.5616	9.3454	12.9072	64.6594
4	7	Ctrl	7.7392	89.8630	46.3648	425.9290	11.0577	10.7178	70.9125
4		5%	6.9783	94.2773	43.2668	423.4370	10.4858	11.1741	72.2514
4		7%	6.8106	97.4097	43.5623	422.4052	10.5589	11.5568	74.6348
4		10%	6.8519	97.2821	43.0786	421.7775	10.9839	11.5360	66.2085
4	15	Ctrl	8.2864	99.7324	36.5363	346.1389	10.6089	8.1230	67.9675
4		5%	7.3720	103.3902	33.7276	315.8497	10.6566	8.8886	67.1840
4		7%	6.3390	95.0281	29.6522	265.5321	10.3426	8.5198	52.8461
4		10%	7.2396	99.4186	32.5943	302.9507	11.0184	8.0860	61.5719
4	30	Ctrl	8.4073	111.9746	36.7387	421.1350	7.9749	11.8792	71.1611
4		5%	8.1726	105.8840	32.9508	408.7692	7.5422	11.3439	66.5609
4		7%	7.8603	109.1092	32.1799	394.3977	8.6721	11.4043	64.1645
4		10%	8.4500	105.9357	34.1709	403.5614	8.8794	12.4357	69.1606

4.4.1 Cobalt

The concentration of Co in each fraction was determined, and the average concentrations of Co in the samples were shown in Figs. 4.11 to 4.15.

In BCR1 fraction (see fig.4.11), all of the samples at 1-, 7-, 15- and 30-day of incubation with different SOM, indicated lower extractability significant differences ($p < 0.05$) of Co as compared to the control. At 1-day of incubation, samples with 7% and 10% SOM showed significant difference ($p < 0.05$) in

extractable Co concentration as compare with control (12.46% and 16.21%, decrease, respectively). There was no significant difference in Co concentration observed in 7-day of incubation samples. At 15-day of incubation, samples with 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Co concentration as compare with control (8.00% and 13.62%, decrease, respectively). At 30-day of incubation, samples with 7% and 10%SOM showed significant difference ($p < 0.05$) in extractable Co concentration as compare with control (7.35% and 16.21%, decrease, respectively).

In BCR 2 fraction (see Fig. 4.12), there was no significant difference of control at 1-day of incubation. At 7- day of incubation, samples with 7% and 10% SOM showed significant differences ($p < 0.05$) in extractable Co concentration as compare with control(8.70% and 14.12% increase, respectively). Significant differences ($p < 0.05$) in Co concentration were also detected among samples for 15-day of incubation and their controls. Samples with 5%, 7% and 10%SOM exhibited 25.03, 50.86% and 30.04% higher extractable Co than that in the control, respectively. At 30-day of incubation, samples with 5% and 7% SOM showed significant differences ($p < 0.05$) in extractable Co concentration as compare with the control (25.03%, and 50.82% increase, respectively). Although, all of the samples containing SOM (i.e. 5%, 7% and 10%) showed higher extractable Co than the control, sample incubated with 7% SOM for 7 days revealed the lowest detectable Co among the samples supplemented with different SOM.

For oxidizable fraction, BCR3 (see Fig. 4.13), Significant differences ($p < 0.05$) in Co concentration were also detected among samples for 1-day of incubation and their controls. Samples with 5% and 7% SOM exhibited 47.77% and 51.21% higher extractable Co than the control, respectively. At 7- and 15-day of incubation, samples with 10% SOM showed significant differences ($p < 0.05$) in extractable Co concentration as compare with control(33.66% and 80.14% increase, respectively). There was no significant difference of control at 30-day of incubation.

For residual fraction (see Fig. 4.14), At 1-day of incubation, samples with 5%, 7% and 10% SOM showed significant differences ($p < 0.05$) in extractable Co concentration as compare with control (15.92%, 22.52% and 12.78% increase, respectively). At 7-day of incubation, samples with 5%, 7% and 10% SOM showed significant differences ($p < 0.05$) in extractable Co concentration as compare with control (9.83%, 12.00% and 11.46% increase, respectively). Significant differences ($p < 0.05$) in Co concentration were also detected among samples for 15 days of incubation and their controls. Samples with 5%, 7% and 10% SOM exhibited 11.04%, 23.50% and 12.63% higher extractable Co, respectively. At 30-day of incubation, there is no significant difference was observed ($p < 0.05$) between Co concentration from samples with addition of SOM and control. (see Fig. 4.14 and Table 4.6).

Regarding of the 3-step BCR sequential extraction and the residual fraction, the percentage distribution of Cobalt in each fraction relatively to the sum of metal concentrations in all fractions (BCR1+ BCR2+ BCR3+ Res), which represent 100%, of all treated soils were determined in all incubation periods (1-, 7-, 15- and 30-day of incubation) (Figure 4.15.) Based on the overall percentage distribution in all incubation periods (1, 7, 15 and 30 days), the percentage of Co in residual fraction (Res) was dominant and the ranking of Co in other fractions is as following;

$$\text{Res (30-40\%)} \approx \text{BCR3 (30-40\%)} > \text{BCR2 (20-25\%)} > \text{BCR1 (10-15\%)}$$

In conclusion, the residual fraction (Res) was the major fraction of Co in this study (the lowest in the % distribution as compared with other fractions). The optimum condition of Co in BCR2 and Residual fraction were 7%SOM. The optimum condition of Co in BCR1 and BCR3 were 10%SOM of soil amendment. However, the addition of 10% and 7%SOM could be the suitable ratio for reducing Co in BCR1.

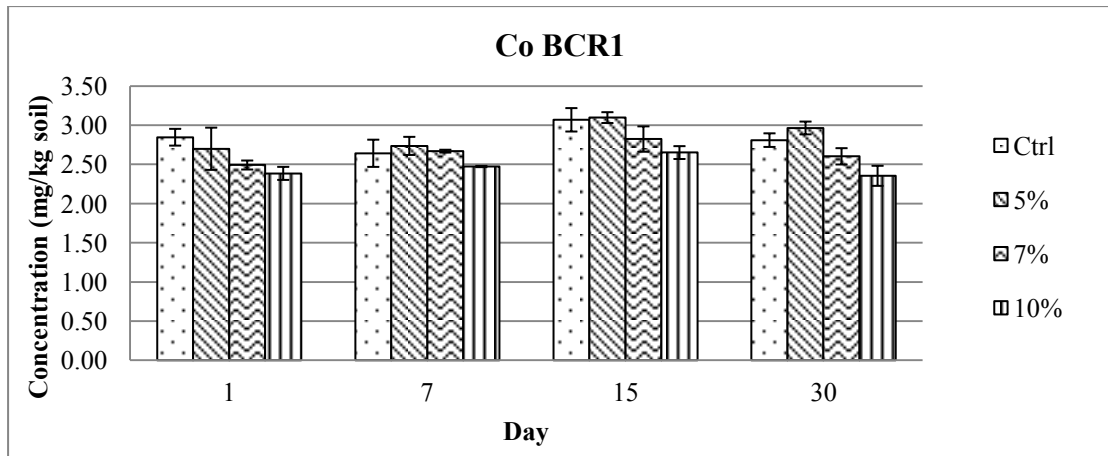


Figure 4.11 Averages Co concentrations in BCR1 within 30 days at different percentages of SOM.

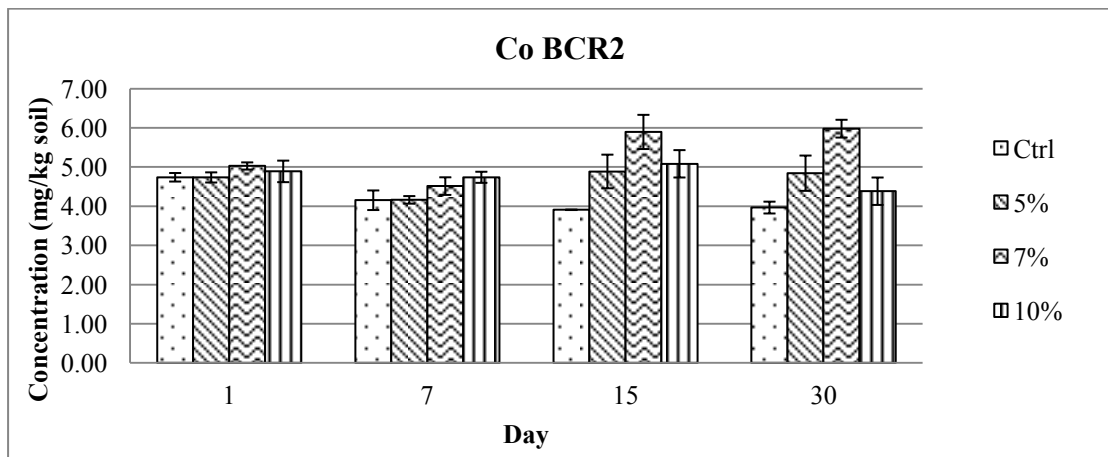


Figure 4.12 Averages Co concentrations in BCR2 within 30 days at different percentages of SOM.

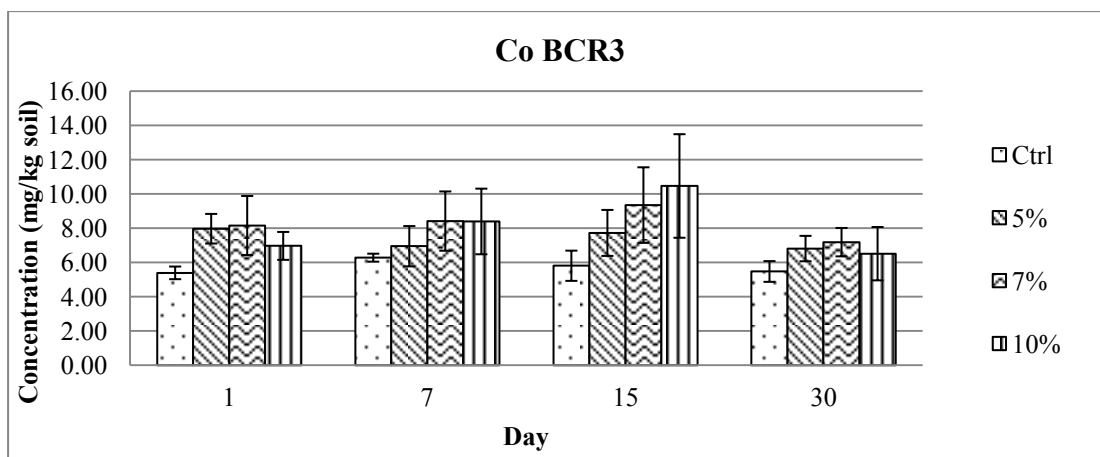


Figure 4.13 Averages Co concentrations in BCR3 within 30 days at different percentages of SOM.

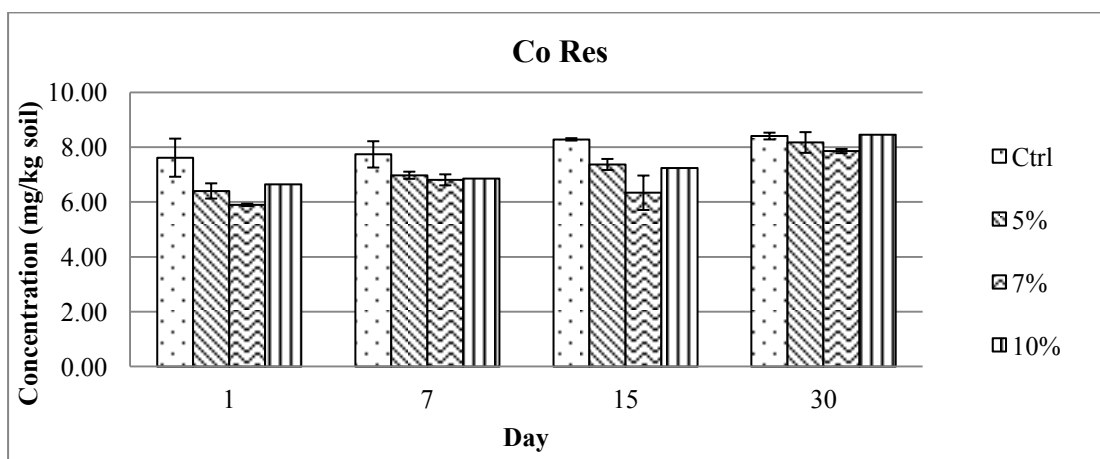


Figure 4.14 Averages Co concentrations in residual fraction within 30 days at different percentages of SOM.

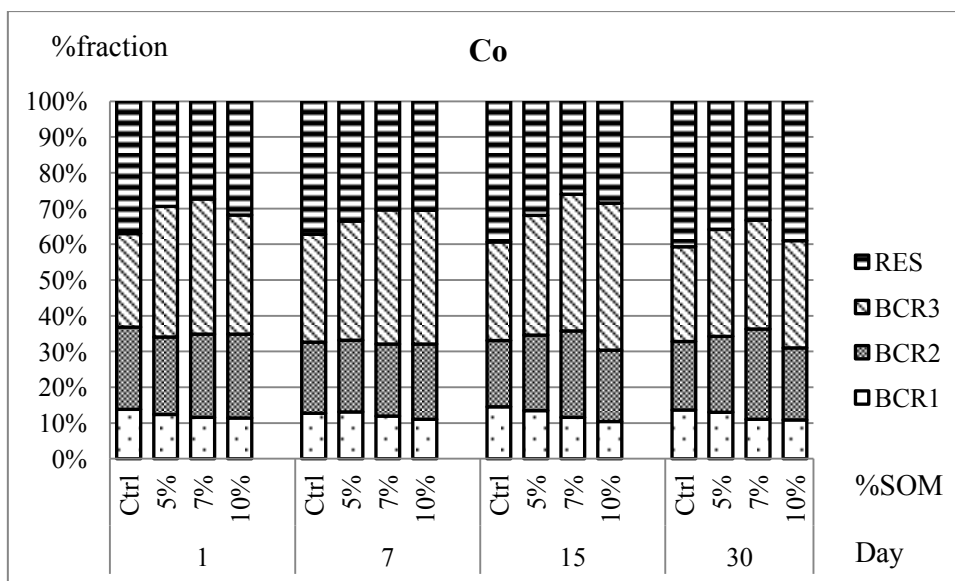


Figure 4.15 All fraction percentages of Co within 30 days at different percentages of SOM.

4.4.2 Chromium

The concentration of Cr in each fraction was determined, and the average concentrations of Co in the samples were shown in Figs. 4.16 to 4.20.

In BCR1 fraction (see Fig. 4.16), all of the samples at 1-, 7-, 15- and 30-day of incubation with different SOM: 5%, 7% and 10% SOM, indicated lower extractability significant differences ($p < 0.05$) of Cr as compared to the control. At 1-day of incubation, there were only samples with 7% SOM, extractable Cr concentration statistically significantly decreased ($p < 0.05$) 26.21%. At 7- and 15-day of incubation, samples with 5% SOM only showed significant differences ($p < 0.05$) in extractable Cr concentration. Comparing to the control, extractable Mn concentration statistically significantly increased ($p < 0.05$) 10.24% and 23.52%, respectively. There was no significant difference ($p < 0.05$) in 30-day-incubation.

In BCR2 fraction (see Fig. 4.17), all of the samples at 1-, 7-, 15- and 30-day of incubation with different SOM: 5%, 7% and 10% SOM, indicated lower extractability significant differences ($p < 0.05$) of Cr as compared to the control. For

1-day of incubation, statistically significantly decreased ($p < 0.05$) 20.33%, 21.96% and 30.59%, respectively. At 7-day of incubation, samples with 5%, 7% and 10% SOM statistically significantly decreased ($p < 0.05$) 9.13%, 13.37% and 17.15%, respectively. At 15-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cr concentration as compare to control (13.48%, 17.51% and 21.94% decrease, respectively). At 30-day of incubation, samples with 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cr concentration as compare with control (14.94% and 16.72% decrease, respectively).

Interestingly, the Cr concentration showed in BCR3 fraction (see Fig. 4.18), of most samples is significantly higher than Cr detected in the controls ($p < 0.05$). Samples were incubated with 5%, 7% and 10% SOM, for 1 day statistically significantly increased ($p < 0.05$) 34.51%, 73.65% and 122.57%, respectively. At 7-day of incubation, samples with 7% and 10% SOM statistically significantly increased ($p < 0.05$) 113.99% and 138.21%, respectively. For 15-day of incubation, There was only found indicated significantly higher extractability ($p < 0.05$) of Cr when compared to the control with 10% SOM (139.99% increase). At 30-day of incubation, samples with 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cr concentration as compare with control (112.73% and 118.02% increase, respectively).

For residual fraction, there was no significant difference ($p < 0.05$) in 1-, 7-, 15- and 30-day of incubation.

For all fraction of Cr (Fig. 4.20), the percentage distribution of Cr in each fraction relatively to the sum of metal concentrations in all fractions (BCR1+ BCR2+ BCR3+ Res), which represent into 100%, of all treated soils were determined in all incubation. Based on the overall percentage distribution in all incubation periods, the percentage of Cr in residual fraction (Res) was dominant and the ranking of Cr in other fractions is as following;

Res (80-85%) > BCR2 (10-15%) > BCR3 (5%) > BCR1 (1-2%)

As mentioned, Almost of Cr was dominantly showed in residual fraction. The extractable Cr in BCR1 and BCR2 fractions were about 15% and time were not effect to Cr. For other research, Ololade (2009) found high concentration of Cu in sewage. Kumar et al., (2011) found heavy metal in Sukinda mining Area and quantity of Cr higher than Cu.

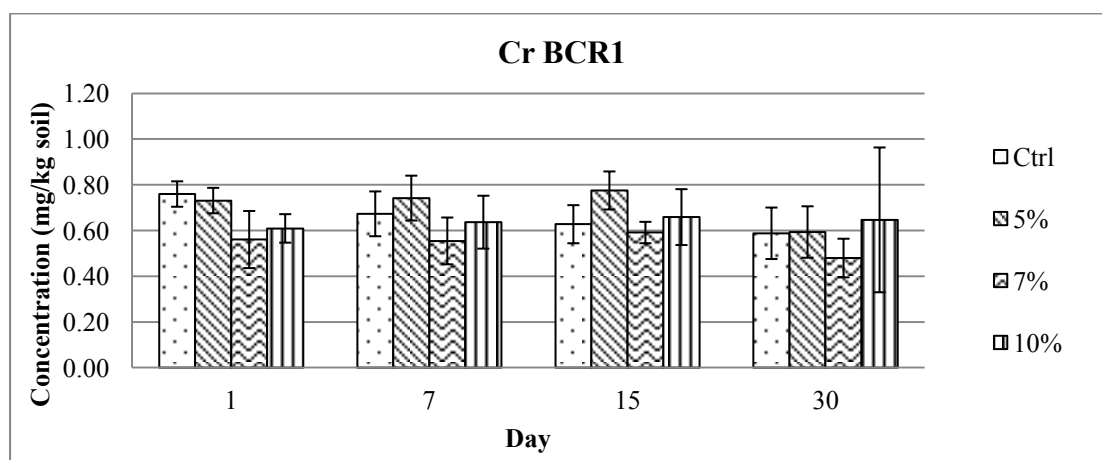


Figure 4.16 Averages Cr concentrations in BCR1 within 30 days at different percentages of SOM.

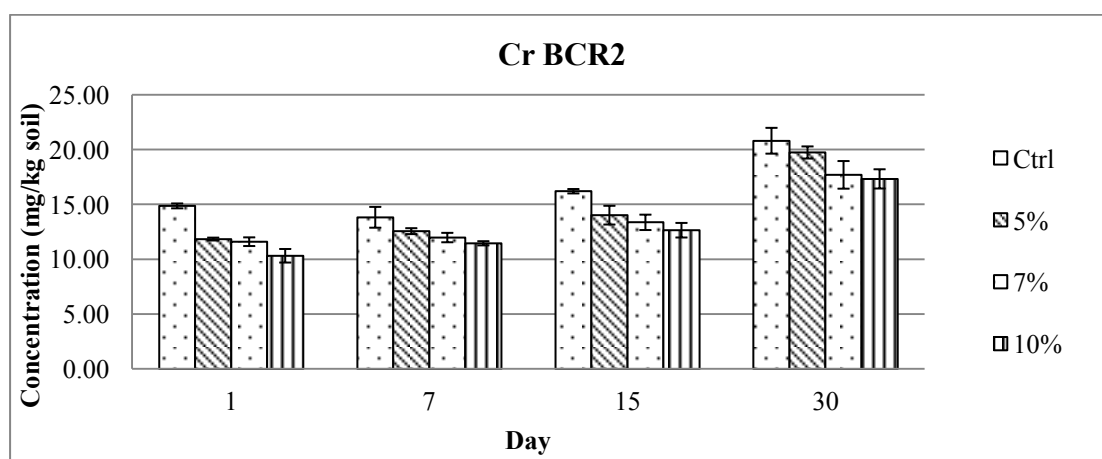


Figure 4.17 Averages Cr concentrations in BCR2 within 30 days at different percentages of SOM.

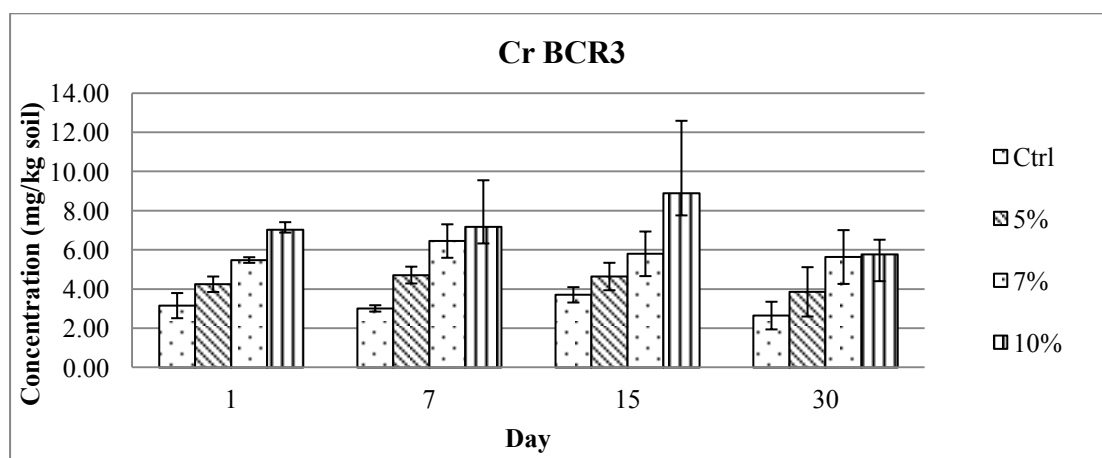


Figure 4.18 Averages Cr concentrations in BCR3 within 30 days at different percentages of SOM.

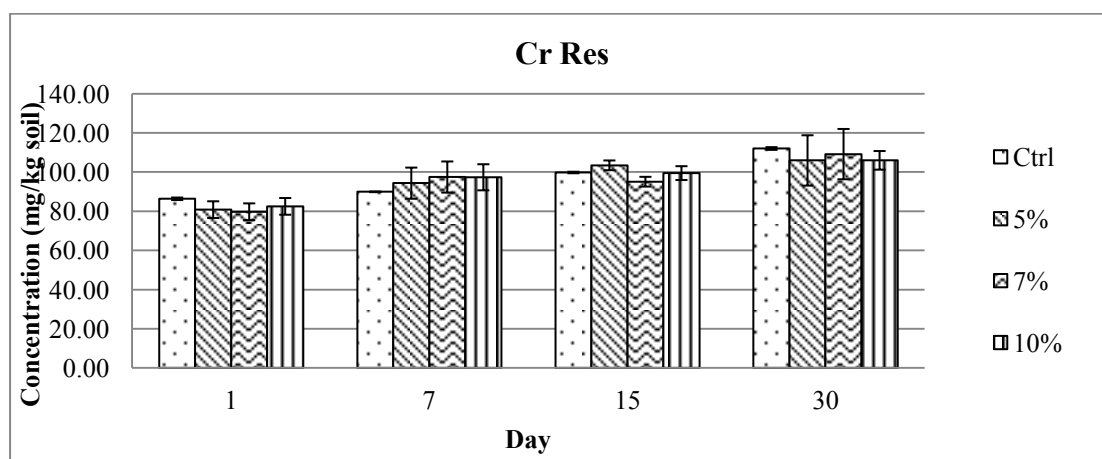


Figure 4.19 Averages Cr concentrations in Residual fraction within 30 days at different percentages of SOM.

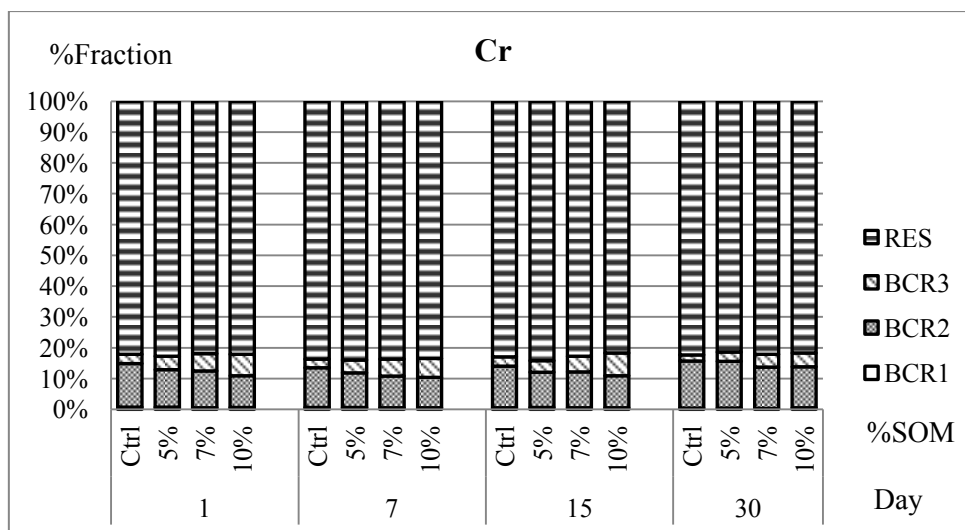


Figure 4.20 All fraction percentages of Cr within 30 days at different percentages of SOM.

4.4.3 Copper

The concentration of Cu in each fraction was determined, and the average concentrations of Cu in the samples are shown in Figs. 4.21-4.24.

After investigated Cu in each fraction, for BCR1 fraction (see Fig. 4.21) all of the samples at 1-, 7-, 15- and 30-day of incubation with different SOM, showed that extractable Cu significantly extraordinary decrease ($p < 0.05$) as compared to those of the control. At 1-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cu concentration as compare to those of the control (85.14%, 88.69% and 91.81% decrease, respectively). . At 7-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cu concentration as compare to those of the control (85.53%, 89.47% and 91.23%, respectively). For 15-dayof incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cu concentration as compare to those of the control (87.12%, 90.17% and 91.50% decrease, respectively). Similarly, for 30-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cu

concentration as compare to those of the control (88.96%, 90.93% and 91.41% decrease, respectively).

In BCR2 fraction (see Fig.4.22), all of the samples at 1-, 7-, 15- and 30-day of incubation with different SOM, indicated significantly lower extractability ($p < 0.05$) of Cu as compared to the control. For 1-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cu concentration as compare with control (57.33%, 75.36% and 81.28% decrease, respectively). For 7 –day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cu concentration as compare to the control (60.49%, 77.08% and 82.35% decrease, respectively). For 15-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cu concentration as compare to the control (64.12%, 74.39% and 84.11% decrease, respectively). For 30-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cu concentration as compare to the control (52.21%, 66.13% and 71.74% decrease, respectively).

In BCR3 fraction (see Fig. 4.23), for 1-day of incubation, samples with 5%, 7% and 10% SOM showed significant differences ($p < 0.05$) in extractable Cu concentration as compare to the control (136.34%, 189.70% and 169.45% increase, respectively) . Significant differences ($p < 0.05$) in Cu concentration were also detected among samples with 7-day of incubation and the control. Samples with 5%, 7% and 10%SOM exhibited 78.25%, 122.54% and 127.37% higher extractable Cu as compare to the control. For 15-day of incubation, samples with 5%, 7% and 10% SOM showed significant differences ($p < 0.05$) in extractable Cu concentration as compare to the control (74.62%, 115.10% and 122.81% increase, respectively). For 30 days incubation, samples with 5%, 7% and 10% SOM showed significant differences ($p < 0.05$) in extractable Cu concentration as compare to the control (135.20%, 153.83% and 155.03% increase, respectively).

For Cu concentration in residual fraction after 3-step sequential extraction (See Fig.4.24), there was no significant difference for Cu concentration of 1-day of incubation but the others for 7-, 15- and 30-day of incubation with different SOM, revealed significantly lower extractability ($p < 0.05$) of Cu as compared to the control. For 7-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cu concentration as compare to the control (6.68%, 6.04% and 7.09% decrease, respectively).. For 15-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Cu concentration as compare to the control (7.69%, 18.84% and 10.79% decrease, respectively). For 30-day of incubation, samples with 5% and 7% SOM showed significant difference ($p < 0.05$) in extractable Cu concentration as compare to the control (10.31%, and 12.41% decrease, respectively).

Regarding of the 3-step BCR sequential extraction and the residual fraction, the percentage distribution of Cu of each fraction, relatively to the sum of metal concentrations in all fractions (BCR1+ BCR2+ BCR3+ Res, which represent totally 100%, of all treated soils were determined in all incubation periods (i.e., 1, 7, 15 and 30 days) as showed in Figure 4.25. Based on the overall percentage distribution in all incubation periods, the percentage of Cu in residual fraction (Res.) was dominant and the ranking of Cu in each fraction was shown as the following order:

$$\text{Res (65-70\%)} > \text{BCR3(15-25\%)} > \text{BCR2(5-8\%)} > \text{BCR1(1-2\%)}$$

For all fractions of extractable Cu concentration, the Cu concentration among %SOMs and the control for BCR1 and BCR2 were significantly decreased. It showed that almost available Cu in BCR1 and BCR2 (Fig. 4.21 and 4.22) were not easily leached into the environment. They were showed the evidence of increasing Cu concentration in BCR3 (see Fig. 4.23). The results showed high concentration in residual fraction as same as research of Janoš et al., (2010). Although the most

fraction of Cu were in residual fraction, when time went on the extractable Cu were found. Results were support by research of Sprynskyy (2006).

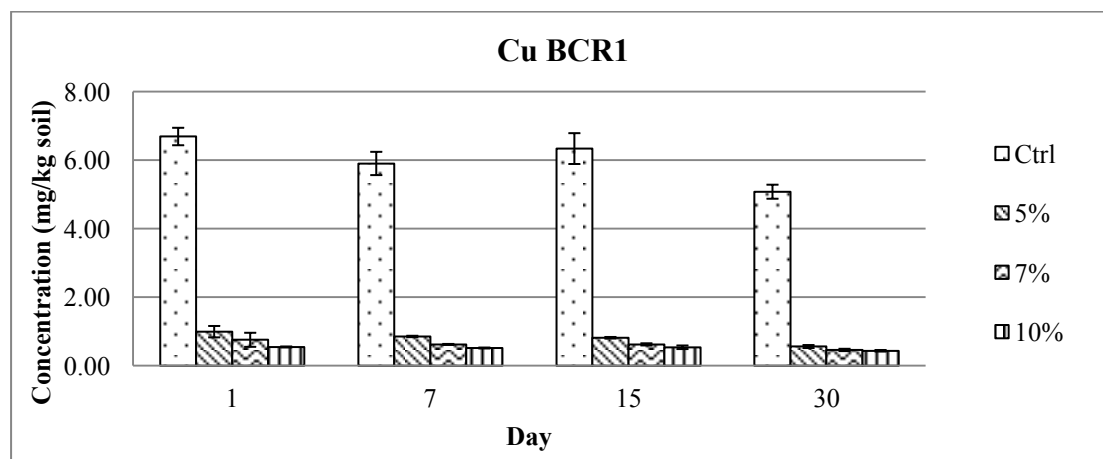


Figure 4.21 Average Cu concentrations in BCR1 within 30 days at different percentages of SOM.

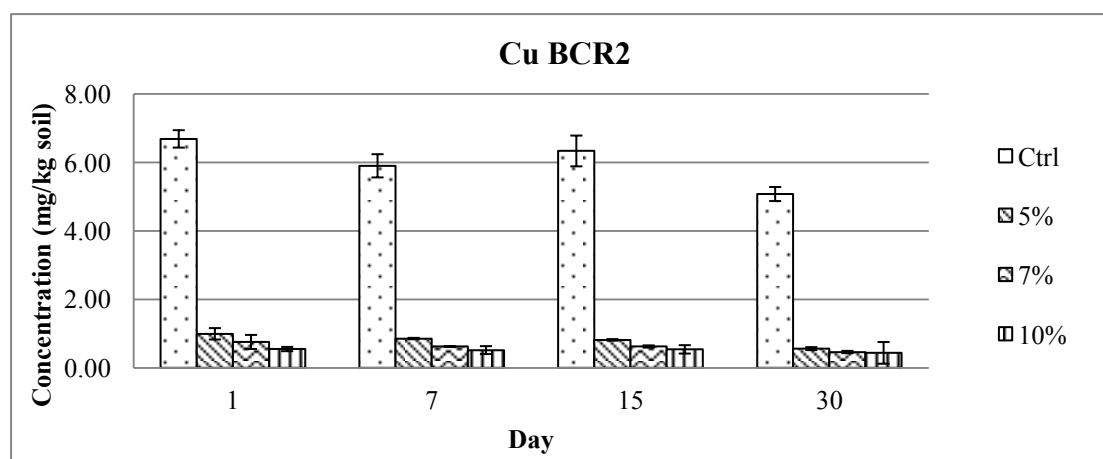


Figure 4.22 Average Cu concentrations in BCR2 within 30 days at different percentages of SOM.

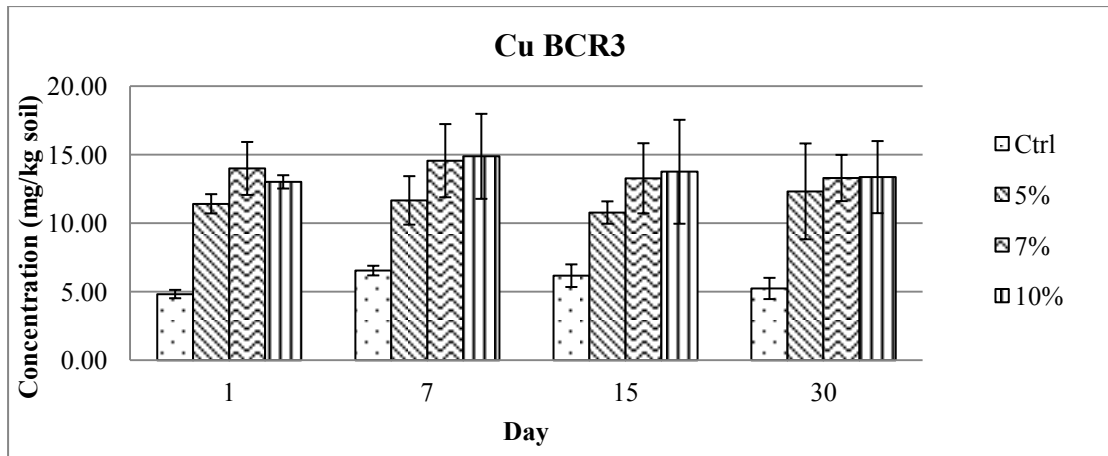


Figure 4.23 Average Cu concentrations in BCR3 within 30 days at different percentages of SOM.

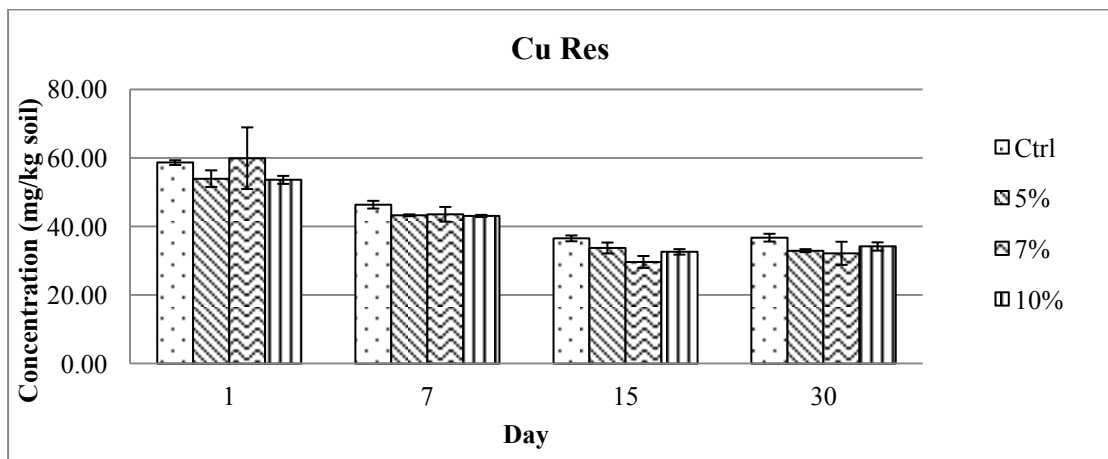


Figure 4.24 Average Cu concentrations in residual fraction within 30 days at different percentages of SOM.

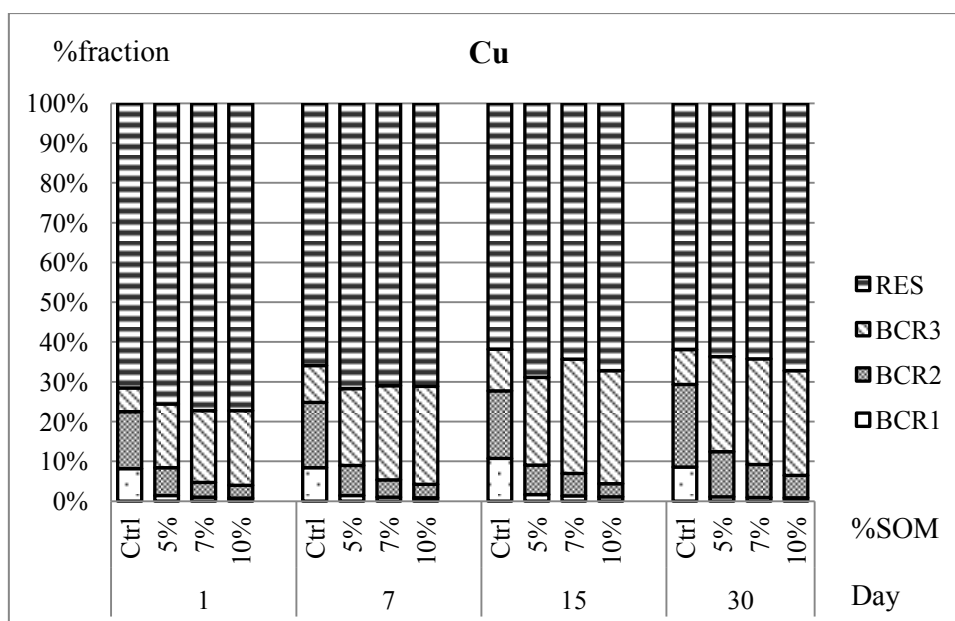


Figure 4.25 Percentages of Cu in all fractions within 30 days at different percentages of SOM.

4.4.4 Manganese

The concentration of Mn in each fraction was determined, and the average concentrations of Mn in the samples are shown in Figs. 4.26-4.29.

In the BCR1 fraction (see Fig.4.26), no reduction in extractable Mn was observed among samples with the addition of 5%, 7%, and 10% of SOM. There is no significant difference was observed ($p < 0.05$) between Mn concentration from samples with addition of SOM and control for 1-day of incubation. At 7-day of incubation, samples with 5%, 7% and 10% SOM showed significant differences ($p < 0.05$) in extractable Mn concentration as compare with control (12.85%, 15.69% and 13.07% increase, respectively). Significant differences ($p < 0.05$) in Mn concentration were also detected among samples for 15 days of incubation and their controls. Samples with 5% and 7% SOM exhibited 12.84% and 9.77% higher extractable Mn respectively. At 30-day of incubation, samples with 5%, 7% and 10% SOM showed significant differences ($p < 0.05$) in extractable Mn concentration as compare with the control (16.65%, 10.33% and 9.78% increase, respectively). Although, all of the

samples containing SOM (i.e. 5%, 7% and 10%) showed higher extractable Mn as compare with their control, sample incubated with 7% SOM for 15 days revealed the lowest detectable Mn among the samples supplemented with different SOM.

In BCR2 fraction (see Fig. 4.27), all of the samples at 1-, 7-, 15- and 30-day of incubation with different SOM, indicated lower extractability significant differences ($p < 0.05$) of Mn as compared to the control. At 1-day of incubation, samples with 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Mn concentration as compare with control (7.59% and 10.29% decrease, respectively). . At 7-dayof incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Mn concentration as compare to control (8.43%, 9.20% and 7.64% decrease, respectively). For 15- and 30-day of incubation, There were only found indicated significantly lower extractability ($p < 0.05$) of Mn when compared to the control with 10% SOM (10.03% and 9.76% decrease, respectively).

Interestingly, the Mn concentration observed in BCR3 fraction (see Fig.4.28) of most samples is significantly higher than Mn detected in the controls ($p < 0.05$). Samples were incubated with 5%, 7% and 10%, for 1 day statistically significantly increased ($p < 0.05$) 61.97%, 107.15% and 104.86%, respectively. At 7-day of incubation, samples with 5%, 7% and 10% SOM statistically significantly increased ($p < 0.05$) 55.56%, 89.44% and 113.29%, respectively. For 15-day-incubation samples with 5%, 7% and 10% SOM statistically significantly increased ($p < 0.05$) 99.98%, 151.64% and 186.17%%, respectively. At 30-day of incubation, samples with 5%, 7% and 10% SOM showed significant differences ($p < 0.05$) in extractable Mn concentration. Comparing to the control, extractable Mn concentration statistically significantly increased ($p < 0.05$) 48.12%, 73.79% and 92.60%, respectively.

For Mn content in the soil residue after BCR extraction (see Fig.4.29), there was no significant difference in Mn concentration observed in 30-day of

incubation samples. Lower concentration of Mn was found in samples incubated with SOM for 1- and 15-day of incubation. After 1-day of incubation, the lowest significant different in Mn concentration was 7% (10.24%, increase). There was no significant difference ($p < 0.05$) in 7-day-incubation. Samples with 5%, 7%, and 10% SOM for 15 days exhibited 8.75%, 23.29% and 12.48%, respectively, lower than that in the control.

The amount of Mn extracted from each fraction and the Mn concentrations contained in the residue were shown in Fig. 4.30. The largest partition of the extractable Mn found in BCR2 fraction was a little higher than that in BCR1. The lowest amount of Mn was detected in BCR3 fraction. Furthermore, the proportion of BCR1 and BCR2 were approximately 70-80% of all fractions and can be concluded as the following order:

$$\text{BCR2 (40-45\%)} > \text{BCR1 (35-40\%)} > \text{Res (20-25\%)} > \text{BCR3(1-3\%)}$$

The results of BCR1 and BCR2 fractions were consistent with the previous studies at Akara mining site, conducted by Chotpantararat et al., (2008), and Changul et al., (2008), they found that Mn is the most metal concentrations leached from batch and column desorption tests.

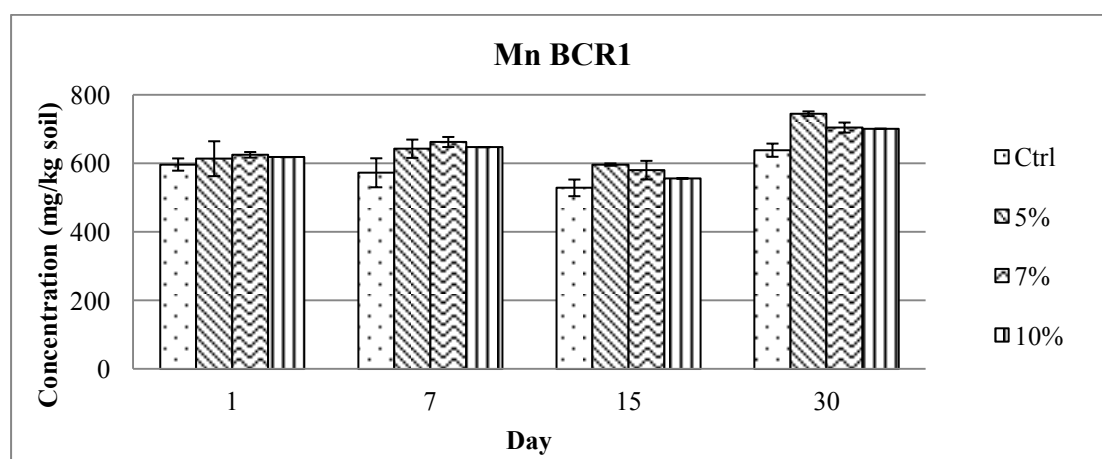


Figure 4.26 Average Mn concentrations in BCR1 within 30 days at different percentages of SOM.

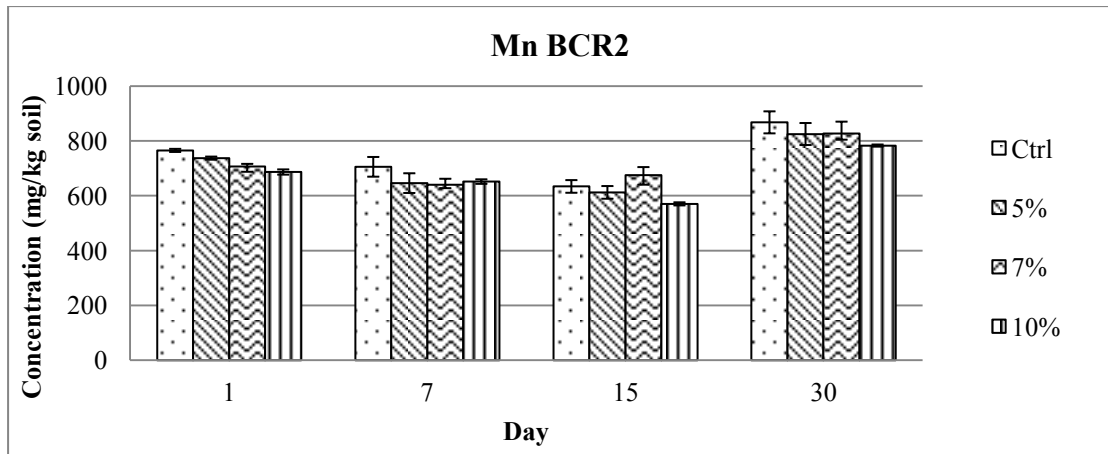


Figure 4.27 Average Mn concentrations in BCR2 within 30 days at different percentages of SOM.

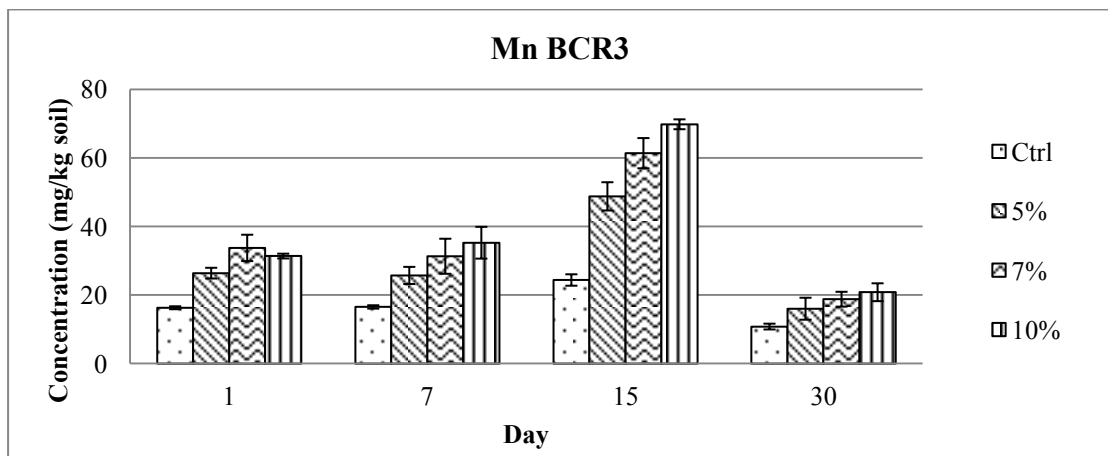


Figure 4.28 Average Mn concentrations in BCR3 within 30 days at different percentages of SOM.

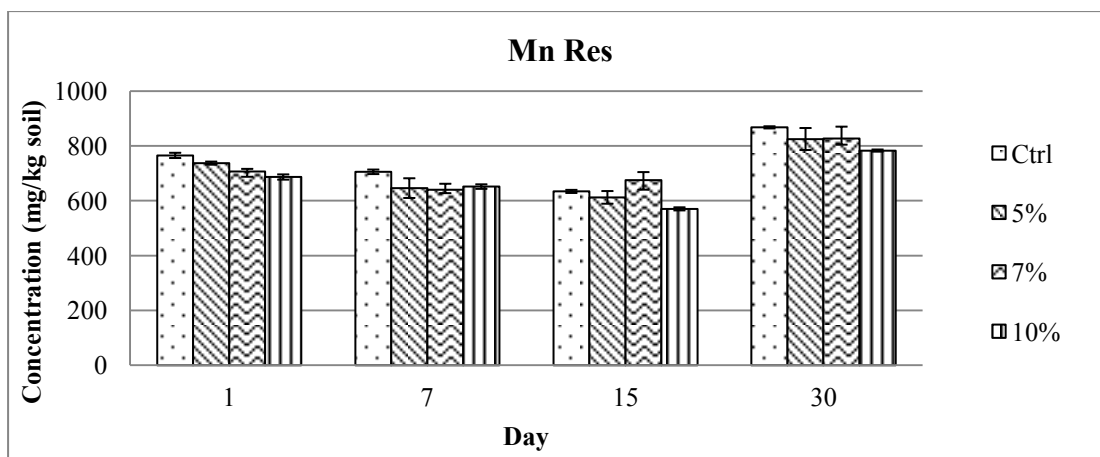


Figure 4.29 Average Mn concentrations in residual fraction within 30 days at different percentages of SOM.

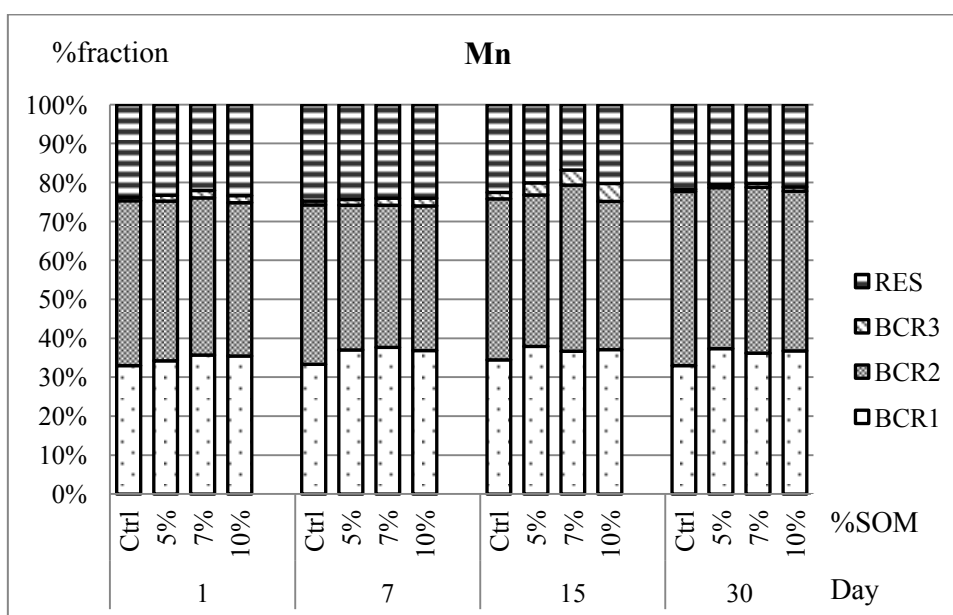


Figure 4.30 All fraction percentages of Mn within 30 days at different percentages of SOM.

4.4.5 Nickel

In BCR1 fraction (see Fig. 4.31), Samples were incubated with 5%, 7% and 10% SOM, for 1-day of incubation Ni statistically significantly increased ($p < 0.05$) 42.66%, 78.19% and 45.30%, respectively. At 7-day of incubation, samples with 5%, 7% and 10% SOM statistically significantly increased ($p < 0.05$) 33.12%, 38.36% and 37.61%, respectively. For 15-day-incubation samples with 5%, 7% and 10% SOM statistically significantly increased ($p < 0.05$) 27.28%, 54.85% and 38.67%, respectively. At 30-day of incubation, samples with 5%, 7% and 10% SOM showed significant differences ($p < 0.05$) in extractable Ni concentration. Comparing to the control, extractable Ni concentration statistically significantly increased ($p < 0.05$) 33.18%, 39.86% and 40.43%, respectively.

In BCR2 fraction (see Fig. 4.32), At 1- day of incubation, samples with 5%, 7% and 10% SOM statistically significantly increased ($p < 0.05$) 9.89%, 21.62% and 21.29%, respectively. At 7-day of incubation, samples with 5%, 7% and 10% SOM statistically significantly increased ($p < 0.05$) 33.12%, 38.36% and 37.61%, respectively. For 15-day-incubation samples with 5%, 7% and 10% SOM statistically significantly increased ($p < 0.05$) 17.23%, 31.58% and 26.49%, respectively. At 30-day of incubation, samples with 5%, 7% and 10% SOM showed significant differences ($p < 0.05$) in extractable Ni concentration. Comparing to the control, extractable Ni concentration statistically significantly increased ($p < 0.05$) 37.35%, 40.88% and 43.19%, respectively.

In BCR3 fraction (see Fig. 4.33), At 1- day of incubation, samples with 5%, 7% and 10% SOM statistically significantly increased ($p < 0.05$) 52.08%, 85.03% and 72.25%, respectively. At 7-day of incubation, samples with 7% and 10% SOM statistically significantly increased ($p < 0.05$) 77.67% and 95.75%, respectively. For 15-day-incubation samples with 5% and 10% SOM statistically significantly increased ($p < 0.05$) 10.08% and 9.82%, respectively. There was no significant difference ($p < 0.05$) in 30-day of incubation.

For residual fraction (see Fig. 4.34), there was only samples with 5% SOM statistically significantly increased ($p < 0.05$) at 15-day of incubation.

The amount of Ni extracted from each fraction and the Ni concentrations contained in the residue were shown in Fig. 4.35. The largest partition of the extractable Ni found in residual fraction. The lowest amount of Ni was detected in BCR1 and BCR3 fraction. Furthermore, the proportion of BCR1 and BCR2 were approximately 30-40% of all fractions and can be concluded as the following order:

$$\text{Res (45-55\%)} > \text{BCR2 (25\%)} > \text{BCR1} \approx \text{BCR3 (8-10\%)}$$

In conclusion, the residual fraction (Res) is the major fraction of Ni in this study. Sprynskyy et al., (2006) also found a decreasing extractable Ni in residual fraction.

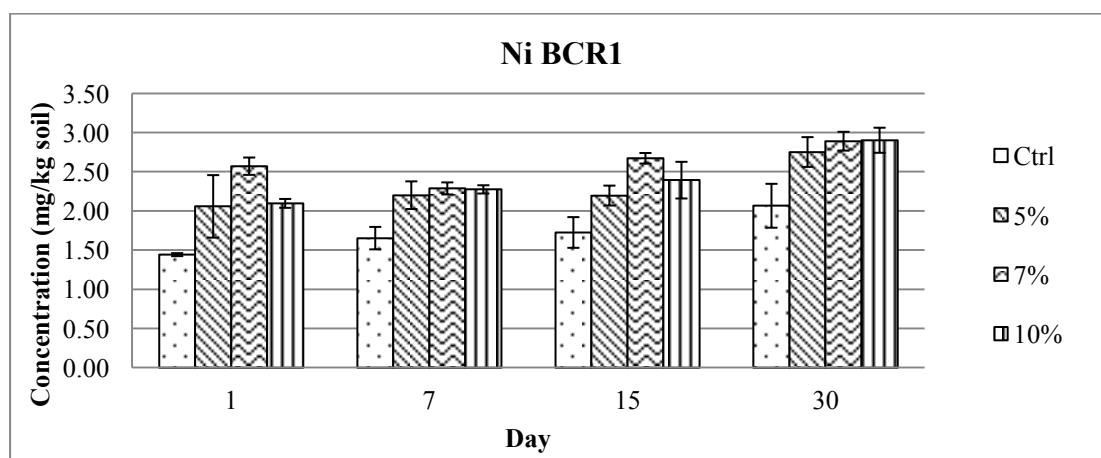


Figure 4.31 Average Ni concentrations in BCR1 within 30 days at different percentages of SOM.

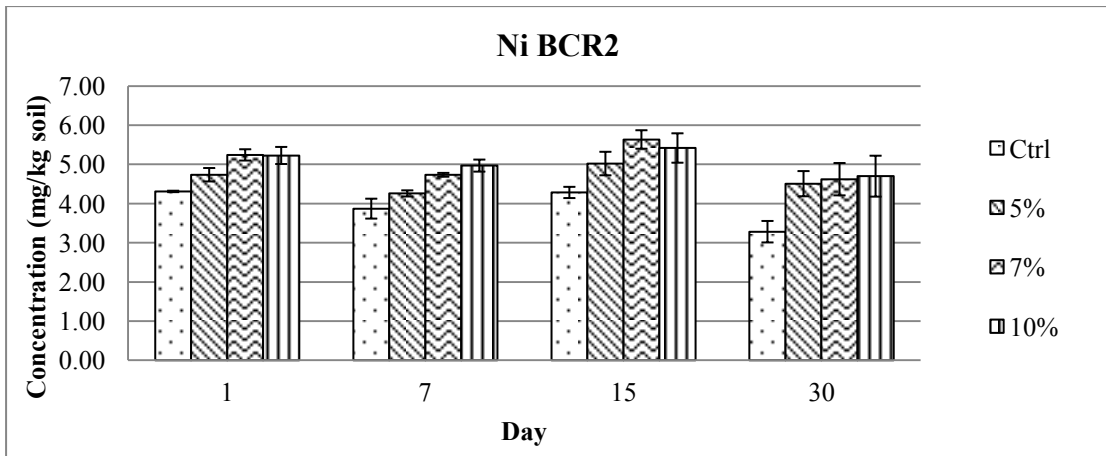


Figure 4.32 Average Ni concentrations in BCR2 within 30 days at different percentages of SOM.

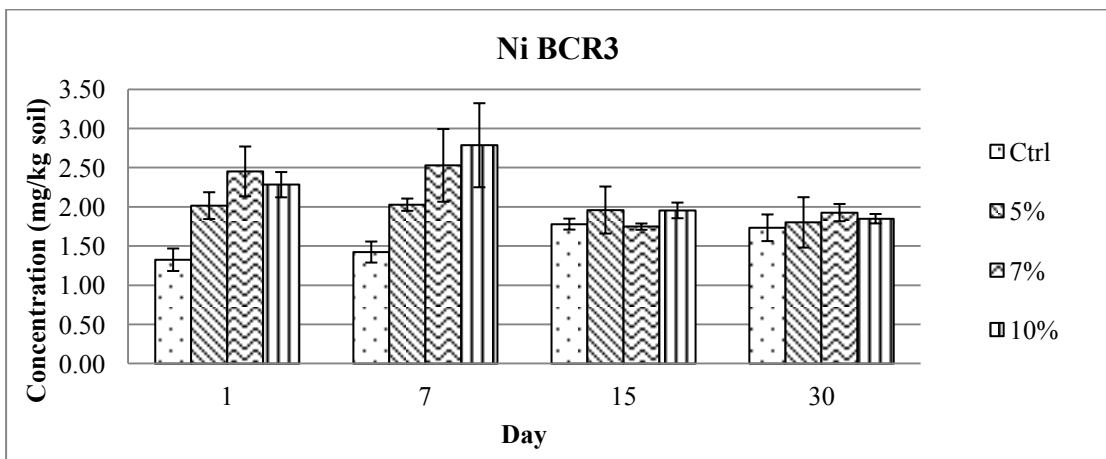


Figure 4.33 Average Ni concentrations in BCR3 within 30 days at different percentages of SOM.

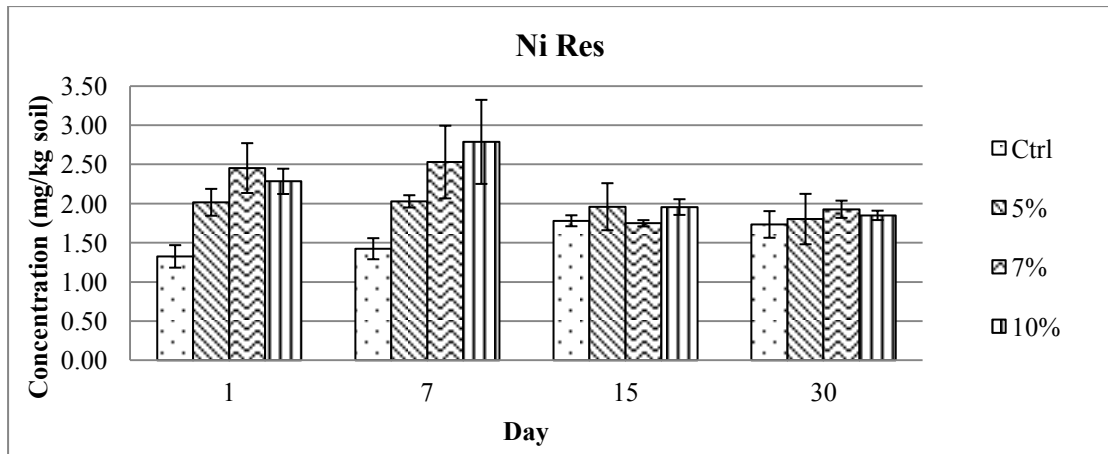


Figure 4.34 Average Ni concentrations in residual fraction within 30 days at different percentages of SOM.

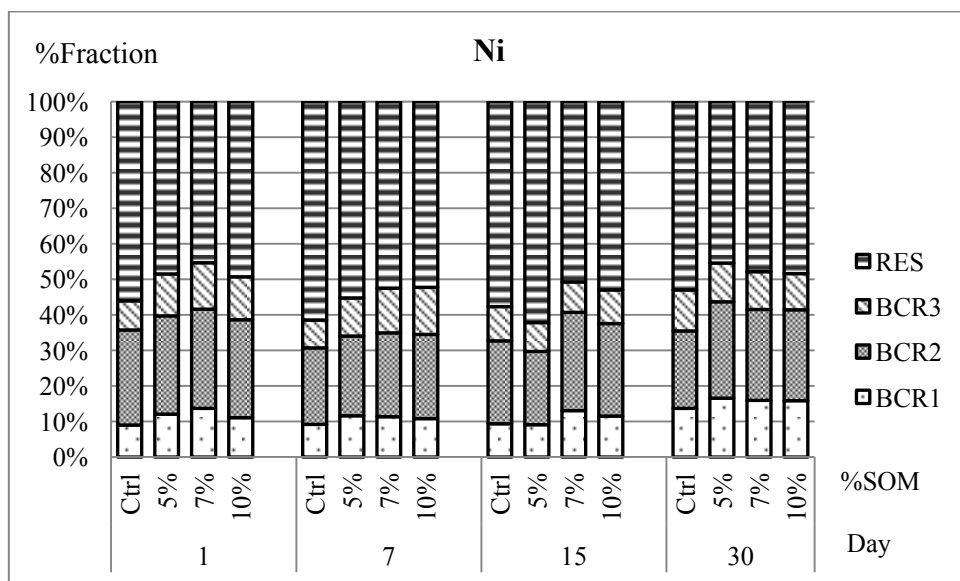


Figure 4.35 All fraction percentages of Ni within 30 days at different percentages of SOM.

4.4.6 Lead

The concentration of Pb in each fraction was determined, and the average concentrations of Pb in the samples are shown in Figs. 4.36-4.39.

In BCR1 fraction (see Fig.4.36), all of the samples at 1-, 7-, 15- and 30-day of incubation with different SOM, found that extractable Pb leached significantly lower than those extractable Pb as compared to the control with significant differences ($p < 0.05$). At 1-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Pb concentration as compared with the control (48.83%, 69.14% and 72.27% decrease, respectively). For 7-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Pb concentration as compared with the control (45.81%, 68.39% and 84.51% decrease, respectively).. For 15-day of incubation with 7% SOM, there was only found indicated significantly lower those in the control ($p < 0.05$) about 53.59%. At 30-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Pb concentration as compared with the control (54.41%, 64.43% and 75.99% decrease, respectively).

In BCR2 fraction (see Fig.4.37), all of the samples at 1-, 7-, 15- and 30-day-incubation with different SOM, indicated higher extractability significant differences ($p < 0.05$) of Pb as compared to the control. At 1-day of incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Pb concentration as compared with the control (7.24%, 18.05% and 24.46% decrease, respectively). At 7-day of incubation, samples with 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Pb concentration as compared with the control (14.02% and 17.22% decrease, respectively). For 15-day of incubation with 10% SOM, there was only found that it decreased significantly lower extractability ($p < 0.05$) of Pb as compared to the control about 14.44%. Furthermore, there was no significant difference in Pb concentration observed in sample incubated with all contents of SOM for 30-day-incubation.

The Pb concentration determined in BCR3 (see Fig.4.38) fraction most SOM containing samples is significantly higher than Pb detected in the controls significant differences ($p < 0.05$). All of the samples at 1-, 7-, 15- and 30-day of incubation with different SOM, indicated significantly higher extractability significant differences ($p < 0.05$) of Pb as compared to those of the control. For 1-day of incubation, samples with 5%, 7% and 10% SOM statistically significant increased ($p < 0.05$) of extractable Pb concentration as comparing to the control approximately 55.16%, 61.17% and 96.21%, respectively. For 7 days incubation, samples with 5%, 7% and 10% SOM statistically significant increased ($p < 0.05$) ($p < 0.05$) of extractable Pb concentration as compare to those of the control approximately 98.19%, 109.12% and 132.57%, respectively. For 15-day of incubation, samples with 5%, 7% and 10% SOM statistically significant increased ($p < 0.05$) of extractable Pb concentration as compare to those of the control approximately 84.11%, 90.15% and 146.78%, respectively. For 30-day of incubation with 10% SOM, there was only found that extractable Pb significantly decreased ($p < 0.05$) as compared to those of the control approximately 71.39%.

In residual fraction (see Fig 4.39), there was no significant differences ($p < 0.05$) in extractable Pb for 30-day of incubation. It probably means that no significant effect of time and %SOM on residual fraction of Pb.

The amount of Pb extracted from each fraction and the Pb concentrations contained in the residue were shown in (Fig. 4.40). The largest partition of the extractable Pb was found in BCR2 fraction. The Lowest amount of Pb was detected in BCR1 fraction. The proportion of all fractions can be concluded from as the following order:

$$\text{BCR2 (50-60\%)} > \text{RES (20-35\%)} > \text{BCR3(15-19\%)} > \text{BCR1(1-2\%)}$$

As mentioned, Pb was easily dissolved as shown higher than 50% of summation of extractable Pb in BCR1 and BCR2 fractions. From the research of Thilo (2008), Pb can be easily dissolved in acidic environment as same as under

reducible condition (BCR2). According to the study of Akkajit and Tongcumpou (2008), metals always dissolve easily in the first and second fractions (BCR1 and BCR2). The level of Pb was in lined with from the research of Chotpantararat (2008) and Channgul (2008) that found Pb in leachate of Akara mine tailings. Furthermore, it may review that a longer duration of incubation time could not effect on extractability of Pb. However, the SOM clearly effect on extractability of Pb on BCR1, BCR2 and BCR3 (see Figs. 4.36-4.38).

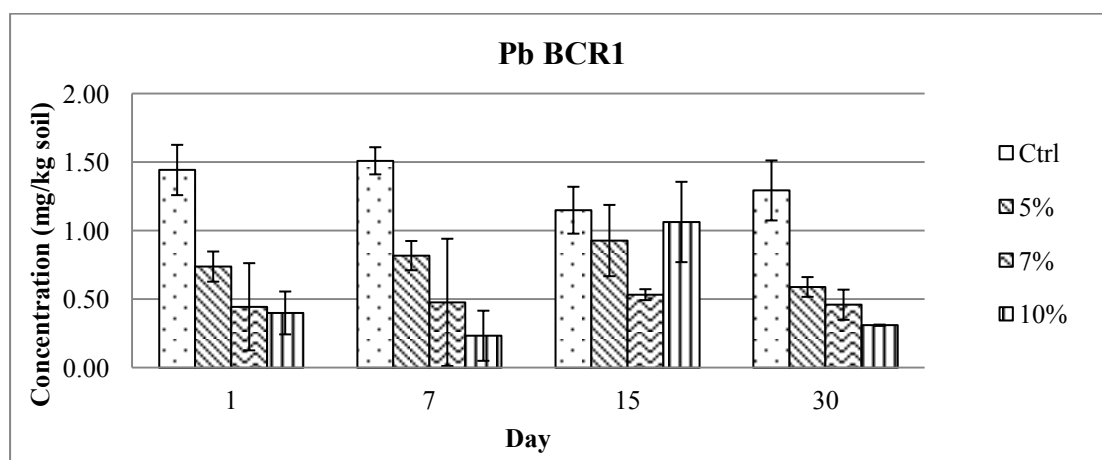


Figure 4.36 Average Pb concentrations in BCR1 within 30 days at different percentages of SOM.

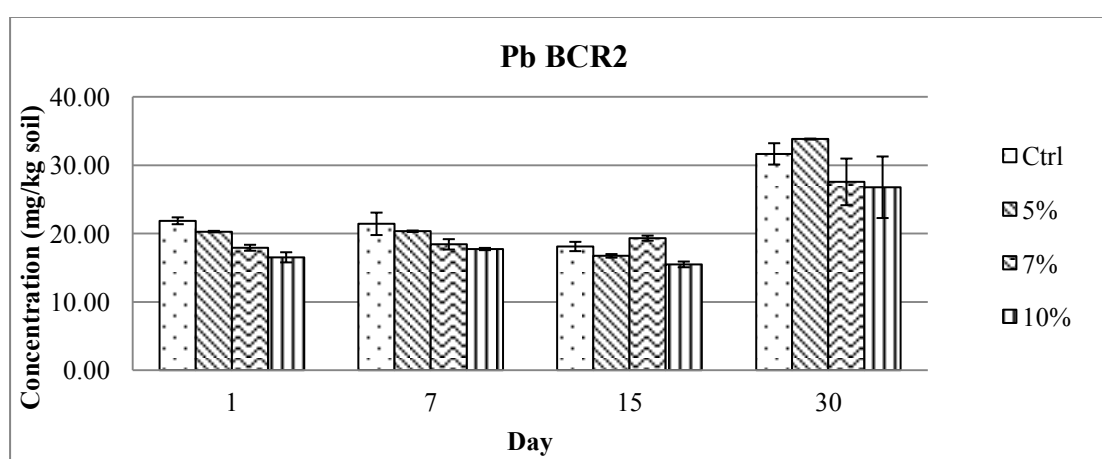


Figure 4.37 Average Pb concentrations in BCR2 within 30 days at different percentages of SOM.

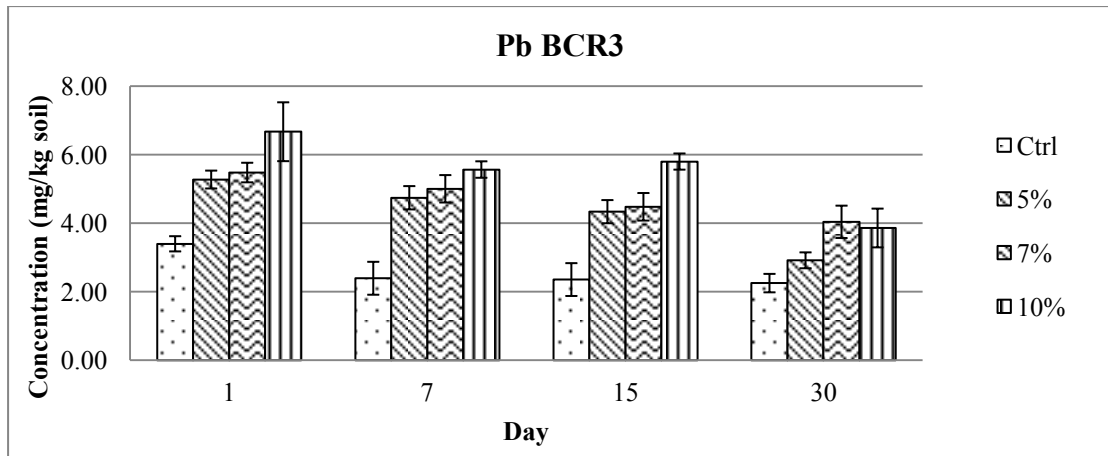


Figure 4.38 Average Pb concentrations in BCR3 within 30 days at different percentages of SOM.

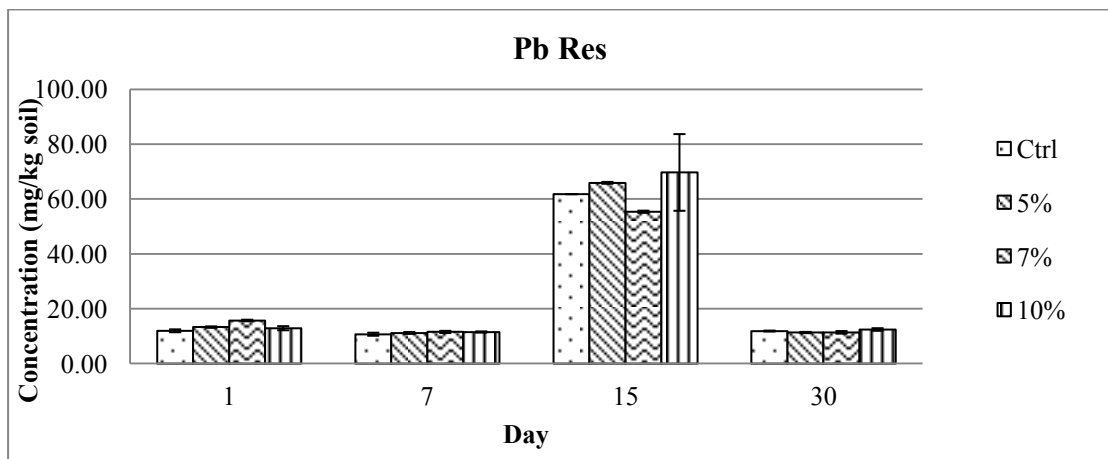


Figure 4.39 Average Pb concentrations in residual fraction within 30 days at different percentages of SOM.

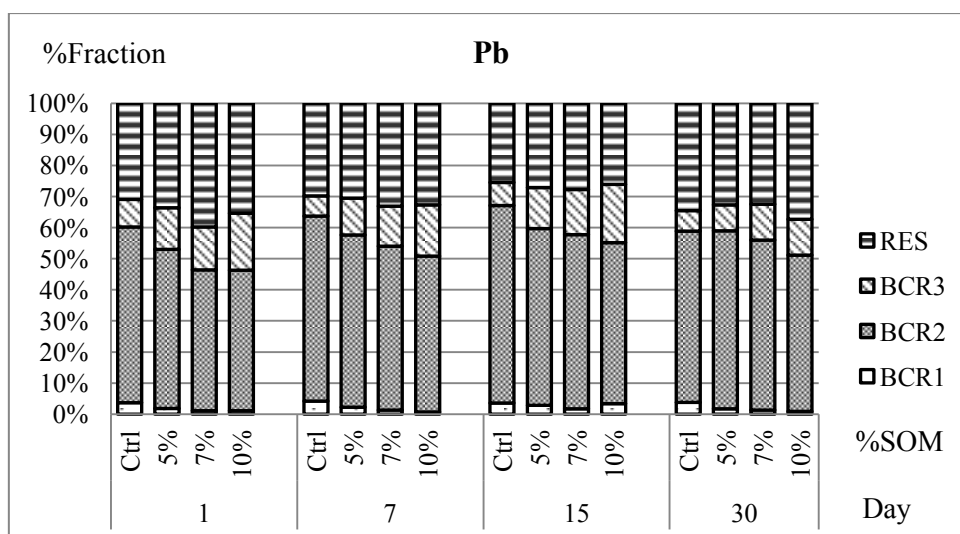


Figure 4.40 All percentage fractions of Pb within 30 days at different percentages of SOM.

4.4.7 Zinc

For BCR1 fraction (see Fig. 4.41), all of the samples at 1-, 7-, 15- and 30-day of incubation with different %SOM, indicated lower extractability significant differences ($p < 0.05$) of Zn as compared to the control. At 1-day of incubation, there was an only significant difference ($p < 0.05$) sample with 7% in extractable Zn concentration as compare with control (16.84% decreases). At 7-day of incubation, samples with 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Zn concentration as compare with control (14.52% and 15.52% decrease, respectively). At 15-dayof incubation, samples with 5%, 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Zn concentration as compare to control (9.49%, 14.50% and 22.01% decrease, respectively). At 30-day of incubation, samples with 7% and 10% SOM showed significant difference ($p < 0.05$) in extractable Zn concentration as compare with control (11.98% and 23.28% decrease, respectively).

For BCR2 fraction (see Fig. 4.42), at 1-day of incubation, samples with 7% and 10% SOM statistically significantly increased ($p < 0.05$) 22.64% and 22.65%, respectively. At 7-day of incubation, samples with 7% and 10% SOM statistically

significantly increased ($p < 0.05$) 36.60% and 26.44%, respectively. For 15-day-incubation samples with 5%, 7% and 10% SOM statistically significantly increased ($p < 0.05$) 17.62%, 39.15% and 21.97%, respectively. For 30-day-incubation samples with 5%, 7% and 10% SOM statistically significantly increased ($p < 0.05$) 84.58%, 28.28% and 21.36%, respectively.

For BCR3 fraction (see Fig. 4.43), at 1-day of incubation, samples with 7% and 10% SOM statistically significantly increased ($p < 0.05$) 29.46% and 44.42%, respectively. At 7-day of incubation, samples with 7% and 10% SOM statistically significantly increased ($p < 0.05$) 48.24% and 64.73%, respectively. At 15-day of incubation, there was only 10% SOM statistically significantly increased ($p < 0.05$) 58.72%. At 30-day of incubation, samples with 7% and 10% SOM statistically significantly increased ($p < 0.05$) 26.67% and 51.89%, respectively.

For residual fraction, There was only 15-day of incubation, which statistically significantly decrease, samples with 7% (22.25%) and 10% (9.41%) SOM.

The percentage of Zn in residual fraction (Res) was dominant and the ranking of Zn in other fractions is as following;

$$\text{Res (70-80\%)} > \text{BCR2 (10-15\%)} > \text{BCR1 (8-12\%)} > \text{BCR3 (5-10\%)}$$

By summing the first-two fraction of BCR sequential extraction (BCR1 and BCR2), Zn in these two fraction is around 20% of the total metal concentration. Changul (2008) and Chotpantararat et al., (2008), Liu et al., (2006) and Jalali et al., (2008) found that as the time increased the Zn in the exchangeable fraction (BCR1) decreased and this is consistent to this study.

In conclusion, the residual fraction was the major fraction of Zn in this study (the lowest in the percentage distribution as compared to other fractions. For BCR1 and Residual fraction, more incubation time should be provided because of no significant difference between the control and other treatments were observed.

However, the addition of 10% of soil amendment (SOM) could be the minimum ratio for Zn in this fraction.

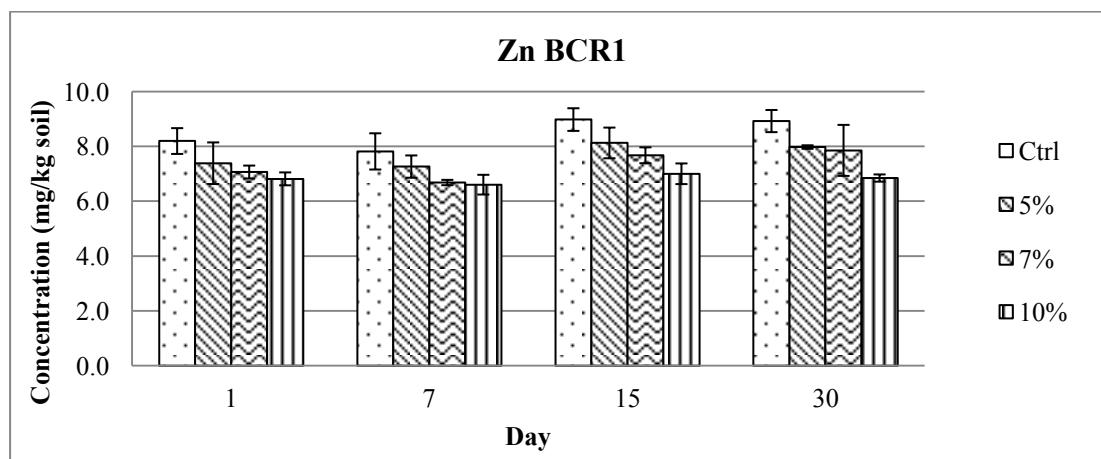


Figure 4.41 Average Zn concentrations in BCR1 within 30 days at different percentages of SOM.

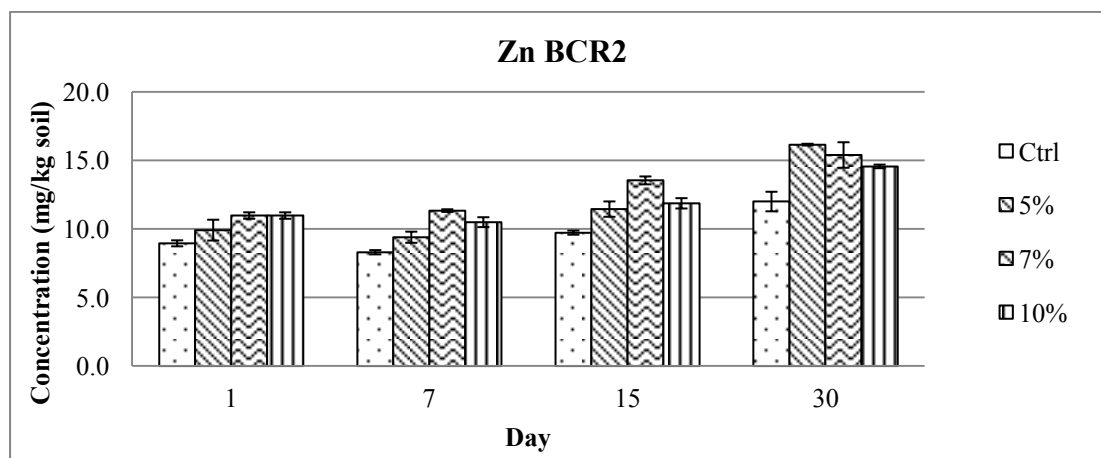


Figure 4.42 Average Zn concentrations in BCR2 within 30 days at different percentages of SOM.

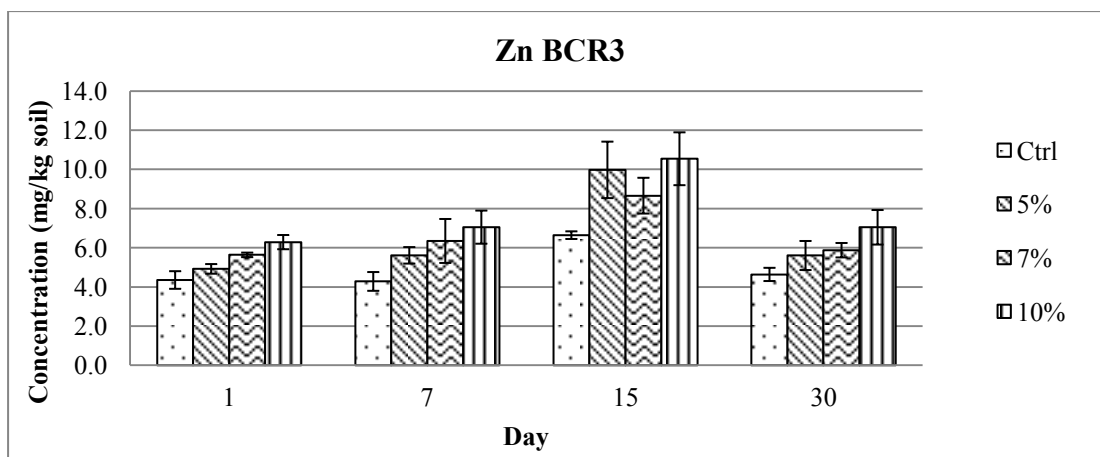


Figure 4.43 Average Zn concentrations in BCR3 within 30 days at different percentages of SOM.

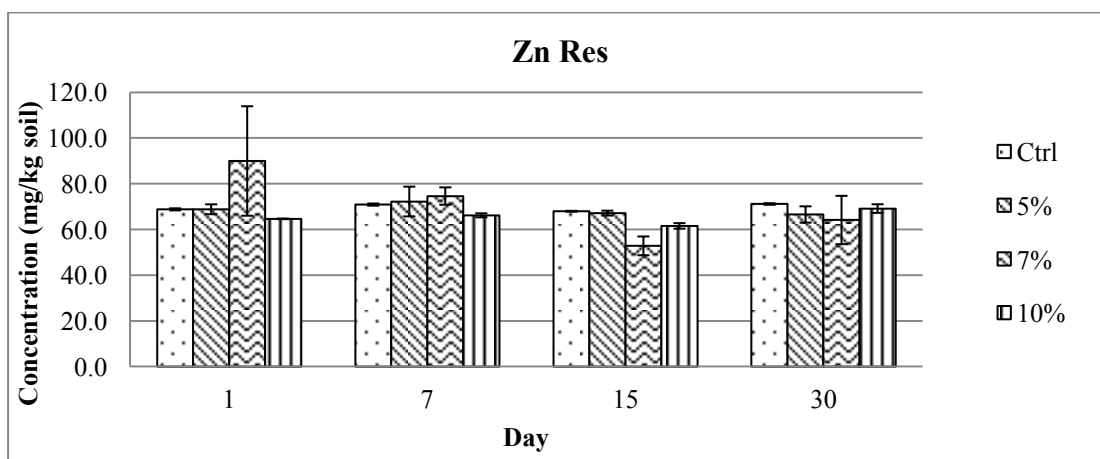


Figure 4.44 Average Zn concentrations in residual fraction within 30 days at different percentages of SOM.

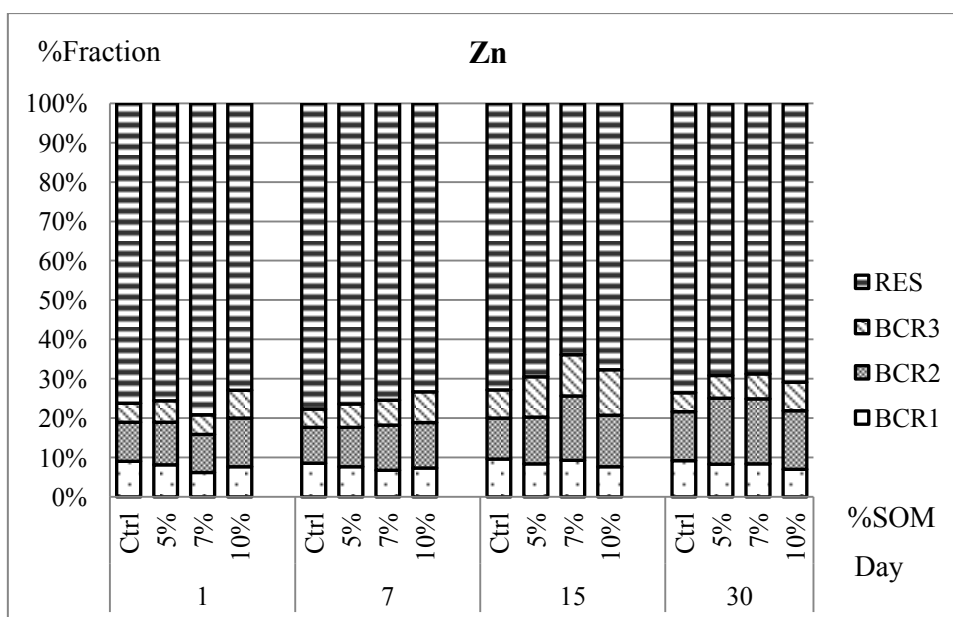


Figure 4.45 All percentage fractions of Zn within 30 days at different percentages of SOM.

4.5 Fractionation patterns of metals

According to ONE-WAY ANOVA, the result showed the increased and decreased trend of elements. However, regression analysis was then selected and used to determine the relationship between incubation times and element. (see Appendix F.). The following equation used semi-log linear regression to describe correlation between time and condition. Details showed as below;

$$\text{Log } Y = a + b \cdot X_1 + c \cdot X_2$$

Where;

a	=	constant
b	=	Coefficient of time
X_1	=	Time
c	=	Coefficient of condition
X_2	=	Condition

Table 4.6 All equations in each metal and fractions from Regression analysis

	BCR1	BCR2	BCR3	RES
Co	Log Y = 1.05 -0.014 X ₁ +0.001X ₂ R²= 0.3645	Log Y = 1.45 + 0.017X ₁ + 0.001X ₂ R²= 0.2272	Log Y = 1.827 + 0.034X ₁ -0.004X ₂ R²= 0.2961	Log Y = 1.957 -0.012X ₁ +0.007X ₂ R²= 0.5589
Cr	Log Y = -0.3072 -0.021X ₁ -0.007X ₂ R²= 0.2167	Log Y = 2.571 + 0.025X ₁ -0.016X ₂ R²= 0.9209	Log Y = 1.167 + 0.084X ₁ - 0.004X ₂ R²= 0.7069	Log Y = 4.450 + -0.001X ₁ - 0.004X ₂ R²= 0.6290
Cu	Log Y = 1.657 -0.255X ₁ -0.012X ₂ R²= 0.8698	Log Y = 2.302 -0.172X ₁ +0.008X ₂ R²= 0.9290	Log Y = 1.84 + 0.092X ₁ -0.0017X ₂ R²= 0.7388	Log Y = 3.982 - 0.009X ₁ - 0.941X ₂ R²= 0.6547
Mn	Log Y = 6.34 + 0.008X ₁ +0.004X ₂ R²= 0.3019	Log Y = 6.536 -0.009X ₁ -0.005X ₂ R²= 0.3028	Log Y = 3.050 + 0.08X ₁ - 0.015X ₂ R²= 0.4515	Log Y = 6.016 - 0.008X ₁ - 0.002X ₂ R²= 0.5077
Ni	Log Y = 0.458 + 0.012X ₁ +0.001X ₂ R²= 0.393	Log Y = 1.43 + 0.028X ₁ -0.004X ₂ R²= 0.5941	Log Y = 0.510 + 0.066X ₁ +0.023X ₂ R²= 0.5785	Log Y = 2.311 + 0.003X ₁ + 0.004X ₂ R²= 0.0600
Pb	Log Y = 0.296 - 0.001X ₁ -0.130X ₂ R²= 0.5353	Log Y = 2.945 - 0.020X ₁ +0.0158X ₂ R²= 0.5893	Log Y = 1.156 - 0.077X ₁ - 0.014X ₂ R²= 0.8434	Log Y = 2.751 - 0.004X ₁ - 2.455X ₂ R²= 0.0156
Zn	Log Y = 2.096 -0.022X ₁ +0.003X ₂ R²= 0.6607	Log Y = 2.128 + 0.024X ₁ +0.013X ₂ R²= 0.779	Log Y = 1.542+ 0.043X ₁ -0.043X ₂ R²= 0.3456	Log Y = 4.2862- 0.006X ₁ -0.003X ₂ R²= 0.0724

According to equations, the negative sign (-) of X₁ and X₂ revealed the significance of elements with %SOM and duration of times (at P <0.05, respectively). For bolded equations mean r-square value were more than 0.70 and also had the negative sign (-) of X₁ and X₂. For BCR1, Cu Zn and Pb were reduced in significance (P <0.05), with Cu and Pb decreased with increasing times in significance (P <0.05). In BCR2, the leaching concentrations of Cu Cr and Pb reduced in significance (P <0.05). But only Ni was affected with time in significance (P <0.05). Moreover, for BCR3, there was no significant differences (P <0.05). with SOM. Time still affected on Cu Cr, Pb and Ni. for residual fraction, condition affected on Cr, Co and Mn. Time also effected on Cu, Co and Mn. Therefore, each equation fraction was concluded and showed in Table 4.7

Table 4.7 Ranking of influence of SOM amount and time aging on reduction of heavy metal leaching

Fraction	Condition (%SOM), X1	Time, X2
BCR1	Reduced leaching rate: Cu, Zn, Pb	Reduced leaching rate: Cu, Pb
BCR2	Reduced leaching rate: Cu, Cr, Pb	Reduced leaching rate: Ni
BCR3	-	Reduced leaching rate: Cu, Cr, Pb, Ni
Res	Reduced leaching rate: Cr, Co, Mn	Reduced leaching rate: Cu, Co, Mn

There are some research for distribution of heavy metal, Brunori et al., (2005) found that in short time, heavy metal can be leached and more soluble than long time incubation and Robinson (1999) said that soil amendment effect or Nickel and Cobalt leaching. Clemente et al., (2006) found that solubility of Mn was increased at the beginning of the experiment due to pH effect. Furthermore, the value of electronegativity (EN) of metals can predict the most strongly and weakly adsorbed by soil. As following: Pb (2.33) > Ni (1.97) > Cu (1.9) > Co (1.88) > Cr (1.66) > Zn (1.65) > Mn (1.55) (Alloway, 1990).

Therefore, most researchers found that Mn were the least adsorbed in the competition situation as same as this study. (Jalali and Moharrami, 2007; Lu and Xu, 2008 and Changul et al., 2010).

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusion

The physicochemical characteristic of samples from ten locations, the average pH of soil tailings was 8.25 and the average pH of humic acid was 2.02. An average percentage of SOM in soil and humic acid were 1.27% and 74.59, respectively.

Tailings from Akara gold mine was determined seven elements; Co, Cr, Cu, Mn, Ni, Pb and Zn. According to three-step BCR and residual fraction, results were shown in percentage of fraction. Almost of higher concentration were found in residual fraction, which were found about 50-60% for Pb, Ni, and about 70-80% in Cr and Cu. However, Mn in BCR1 and BCR2 was found higher than other metals and also Pb was found about 50% in BCR1 and BCR2.

Each heavy metal fraction were investigated under different SOMs (5%, 7% and 10%) and time aging and analyzed by one-way ANOVA and LSD statistical programme. The summary results found that: for BCR1, adding SOM reduced Cu Zn and Pb amount, and as reduced amount of Cu and Pb was reducing time aging ($P < 0.05$). BCR2, Cu Cr and Pb were reduced leaching rate when percentages of SOM were high. And longer time only affected on Ni. For BCR3, there was no significant different in any correlation equations with SOMs. However, time still effected on Co, Cu, Cr, Pb and Ni. Moreover, for residual fraction, condition affected on Cr, Co and Mn. Time also effected on Cu, Cr and Mn. However, when heavy metal were considered by semi-log linear regression. It about highly concentration ($r > 0.7$) of Cu, at BCR1, Cu and Cr at BCR2 and Cu and Cr at BCR3.

5.2 Recommendation

For collecting samples in tailing facilities, it was hard to take a samples in high depths, Because of characteristic and landscape of TSF area almost have watery in the middle.

Manganese correlation in sample was quite high, especially in BCR1 and BCR2. Therefore, it should have a good management to prevent leaching of Mn and other metals to surrounding areas.

According to results, the optimum of SOM (at least 10%) the optimum content of SOM(at least 10%) would be recommend to mining site for mine reclamation. In addition, time aging on such mine tailings will be taken in consideration for reduction heavy metal leaching.

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APPENDICES

APPENDIX A

APPARATUS AND MATERIALS

Table A-1 Apparatus and Materials used in this study

Apparatus	Model	Series Number
1. PNP Orbital Shaker	OS-3	PNP062/48
2. Sartorius Balance 4 digits ,Germany	TE 214S	SWB:17508312
3. Thermolyne Hot Plate stirrer 7x7 inches, USA	SPA-1020B	1138040375412
4. Varian Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES), Australia	VISTA-MPX Axial	EL 02086289
5. Kendro Centrifuge, Germany	Biofuge Statos	40163872
6. Binder Oven, Germany	400(E2)	00-182047
7. Milestone Microwave Digestion and Extraction System, Italy	ETHOS PRO	127547
8. Milestone Microwave Digestion and Extraction System, Italy	ETHOS SEL	125060
9. Thermo Forma Freezer, USA	925	803075-2586
10. Orto alresa Sieve shaker and sieve 200 mesh, 0.075 mm or 75 um, Spain	TA002	542-54/03
12. ELGA Water purification system (18 MΩ), England	Option R7 Maxima HPLC	OR07F 181595BP

Appendix B

Table B-1 The Thailand soil quality standards for habitats and agriculture (PCD, 2008)

parameters	The Thailand soil quality standards for habitats and agriculture
Cr (hexavalent)	<300 mg/kg
Cadmium and compounds	< 37 mg/kg
Lead (Pb)	< 400 mg/kg
Nickel (Ni)	< 1,600 mg/kg
Manganese (Mn)	< 1,800 mg/kg
Arsenic (As)	< 3.9 mg/kg
Mercury and compounds	< 23 mg/kg

Table B-2 The Thailand industrial effluent standard and The standard of surface water quality for agriculture (PCD, 2008)

parameters	The Thailand industrial effluent standard	The standard of surface water quality for agriculture
pH	5.5-9	5-9
Zinc (Zn)	< 0.05 mg/l	< 1.0 mg/l
Cr (hexavalent)	< 0.25 mg/l	<0.05 mg/l
Cr (Trivalent)	< 0.75 mg/l	<0.5mg/l
Copper (Cu)	< 2.0 mg/l	<0.1 mg/l
Cadmium (Cd)	< 0.03 mg/l	< 0.05 mg/l
Lead (Pb)	< 0.2 mg/l	< 0.05 mg/l
Nickel (Ni)	< 1.0 mg/l	<0.1 mg/l
Manganese (Mn)	< 5mg/l	< 1.0 mg/l
Arsenic (As)	< 0.25 mg/l	< 0.01 mg/l

Appendix C

Table C-1. Physicochemical Characteristics of Studies Soil Tailings

Samples	Soil tailings properties	
	pH	OM (%)
1-1	8.08	1.49
1-2	8.00	
1-3	8.20	
2-1	8.22	1.23
2-2	8.20	
2-3	8.14	
3-1	8.26	1.04
3-2	8.15	
3-2	8.28	
4-1	8.34	1.50
4-2	8.31	
4-3	8.29	
5-1	8.34	1.38
5-2	8.37	
5-3	8.30	
6-1	8.40	1.17
6-2	8.42	
6-3	8.25	
7-1	8.19	1.23
7-2	8.17	
7-3	8.31	
8-1	8.38	1.11
8-2	8.45	
8-3	8.22	
9-1	8.25	1.51
9-2	8.29	
9-3	8.09	
10-1	8.15	1.04
10-2	8.19	
10-3	8.15	

Table C-2. Physicochemical Characteristics of humic acid

Samples	OM (%)
H-1	78.33
H-2	70.05
H-3	75.39

Appendix D

Table D-1. Total metal concentrations (Co, Cr, Cu, Mn, Ni, Pb and Zn) in 123 Soil tailings samples and CRM 050-025

Samples	Metal concentration in Samples (mg/Kg)						
	Co	Cr	Cu	Mn	Ni	Pb	Zn
BCR1_1/1	N/A	0.6082	6.4415	475.7109	2.0505	1.7665	18.3444
BCR1_1/2	2.5296	0.5759	6.5220	498.6683	1.7138	1.4363	14.9187
BCR1_1/3	2.4027	0.4543	6.3995	484.4058	1.7694	1.7141	15.0399
BCR1_2/1	2.0804	N/A	N/A	N/A	N/A	N/A	N/A
BCR1_2/2	2.1291	0.4374	6.3294	564.7129	1.4192	1.0196	9.6698
BCR1_2/3	2.1551	0.4416	6.4176	569.6464	1.3737	1.1261	9.4970
BCR1_3/1	2.4314	0.4607	6.9634	590.9913	1.5423	1.0943	13.7836
BCR1_3/2	2.3996	0.4336	6.0467	514.2230	1.5106	0.8927	11.3968
BCR1_3/3	2.8622	0.6313	7.1155	605.4416	2.0838	1.4059	9.9696
BCR1_4/1	2.4510	0.4123	6.9223	592.8656	1.4696	3.7229	12.8841
BCR1_4/2	2.5181	0.4405	6.9929	599.1732	1.5374	2.9616	11.5638
BCR1_4/3	2.3711	0.4115	6.9080	575.2974	1.4095	2.6494	11.6201
BCR1_5/1	1.5400	N/A	5.1264	599.2891	1.2055	3.7966	10.7050
BCR1_5/2	1.6112	N/A	5.3027	598.1807	1.1502	3.9869	10.6585
BCR1_5/3	1.6162	N/A	7.9459	607.2612	1.3905	3.4326	14.8508
BCR1_6/1	N/A	0.5376	6.5319	608.5612	1.9373	1.0514	12.6262
BCR1_6/2	2.2994	0.6031	6.5207	596.9697	1.7641	1.1965	15.6754
BCR1_6/3	2.6859	0.5233	6.6050	621.5940	1.5507	1.3751	17.0597
BCR1_7/1	2.6046	0.4993	7.0667	616.2582	1.5741	1.4237	15.8322
BCR1_7/2	2.8018	0.5772	7.3521	625.6451	1.6793	1.4437	15.2457
BCR1_7/3	2.7795	0.4799	7.2418	616.6864	1.6025	1.4989	15.5014
BCR1_8/1	1.8781	0.5033	6.2938	550.1497	1.4855	1.0041	14.6821
BCR1_8/2	1.8112	0.4444	6.3369	550.3086	1.4442	1.2664	12.5849
BCR1_8/3	1.7225	0.4158	6.2802	461.9763	1.4999	1.1402	12.1621
BCR1_9/1	2.5665	0.4837	7.0297	591.0530	1.6211	2.0716	13.5364
BCR1_9/2	2.5702	0.5529	7.0553	583.4978	1.5674	1.6734	13.2065
BCR1_9/3	N/A	0.4629	6.9760	576.2254	1.6382	1.9121	13.8456
BCR1_10/1	2.6441	0.5358	7.5609	613.1167	1.6401	1.9544	9.5407
BCR1_10/2	2.5967	0.5079	7.7513	625.7485	3.0110	1.7525	13.0966
BCR1_10/3	2.6222	0.5362	7.7184	629.8632	1.6033	1.7684	15.6760
BCR2_1/1	11.7986	13.6319	12.4601	598.3807	4.3645	24.0209	12.8025
BCR2_1/2	12.1542	13.9746	11.5071	577.0622	4.5721	24.7482	12.4743
BCR2_1/3	13.0265	13.9013	11.0486	575.0493	4.4876	26.6415	12.7331
BCR2_2/1	15.2669	12.4956	8.4809	690.7863	3.4715	23.0898	8.8098
BCR2_2/2	16.6287	16.4566	11.1246	783.5486	4.4256	30.2460	10.6541
BCR2_2/3	N/A	16.0763	10.6532	737.8228	4.4565	28.4005	10.6222
BCR2_3/1	15.1070	14.4351	10.7915	680.9992	4.6345	29.5181	11.7604
BCR2_3/2	N/A	14.1392	10.4891	664.6719	4.3858	27.6613	17.7084
BCR2_3/3	12.6297	12.5146	9.2192	578.7066	3.9917	26.3214	9.5162
BCR2_4/1	14.2712	7.6650	5.9034	436.5786	3.0162	24.2684	7.6650
BCR2_4/2	14.0461	13.7953	9.5358	715.7480	4.4291	33.2394	10.0602

Table D-1. Total metal concentrations (Co, Cr, Cu, Mn, Ni, Pb and Zn) in 123 Soil tailings samples and CRM 050-025 (Cont.)

Samples	Metal concentration in Samples (mg/Kg)						
	Co	Cr	Cu	Mn	Ni	Pb	Zn
BCR2_4/3	14.0654	13.8561	9.4445	705.9080	4.4171	32.5456	11.0008
BCR2_5/1	12.3373	12.2468	8.2820	723.4992	3.8317	33.5000	9.9068
BCR2_5/2	12.1163	12.4861	8.2136	690.7257	3.8885	34.9429	9.5480
BCR2_5/3	12.1859	12.3749	8.1081	690.8840	3.7898	33.3781	10.8335
BCR2_6/1	14.3405	14.5018	10.8886	676.9322	4.7051	27.0583	11.6500
BCR2_6/2	14.3266	15.1986	11.2608	709.9282	4.8066	28.3995	11.5112
BCR2_6/3	13.8170	13.9945	10.5905	655.0844	4.5457	27.1955	11.3204
BCR2_7/1	N/A	N/A	N/A	N/A	N/A	N/A	N/A
BCR2_7/2	14.5832	14.3799	11.1207	692.2985	4.7285	28.2803	10.9520
BCR2_7/3	N/A	18.5369	11.9408	714.2406	8.6966	28.9026	96.2170
BCR2_8/1	13.4257	9.2648	6.5975	475.3941	2.9741	19.8627	7.4508
BCR2_8/2	13.8395	15.2005	10.5531	778.3795	4.4551	30.5375	11.7758
BCR2_8/3	14.1656	14.7209	10.5794	774.9012	4.3751	29.9391	10.5937
BCR2_9/1	14.8642	14.3789	10.3420	783.8084	4.4513	32.8401	10.7751
BCR2_9/2	14.2388	14.8168	10.5681	785.2349	4.6913	30.8721	10.1548
BCR2_9/3	14.7636	14.4541	10.1869	793.3717	4.4886	30.7898	10.8847
BCR2_10/1	14.5809	15.0378	11.1440	689.3204	5.0533	31.5340	10.3606
BCR2_10/2	N/A	14.3685	10.6834	653.6527	4.6982	29.2655	10.7589
BCR2_10/3	13.6756	13.9901	11.1366	616.6568	4.4323	22.1309	9.5959
BCR3_1/1	6.5803	N/A	N/A	0.1036	0.9404	3.4226	0.3796
BCR3_1/2	8.7781	N/A	N/A	0.0697	0.5941	2.9021	0.1175
BCR3_1/3	12.0485	N/A	N/A	0.0728	0.7072	2.3590	0.1432
BCR3_2/1	N/A	N/A	N/A	0.0752	0.6006	2.1026	0.1154
BCR3_2/2	N/A	N/A	N/A	N/A	N/A	N/A	N/A
BCR3_2/3	1.3880	3.7684	6.8514	30.4629	2.3524	4.8874	5.7266
BCR3_3/1	1.0883	4.1770	6.7462	33.4840	2.6267	5.0320	6.5583
BCR3_3/2	1.3720	8.1144	17.8132	36.8327	8.3303	7.6227	146.5839
BCR3_3/3	1.0926	4.3062	6.5433	34.5165	2.7908	6.4348	6.4846
BCR3_4/1	1.3139	3.3575	6.4417	36.5816	2.6640	9.8670	6.2525
BCR3_4/2	1.4098	3.1051	6.3765	33.1988	2.9291	9.0871	6.6275
BCR3_4/3	1.4054	3.1916	6.6544	32.6913	3.2293	10.0694	6.7501
BCR3_5/1	1.0160	2.1365	5.0859	24.3562	1.9377	12.0256	5.3273
BCR3_5/2	0.8617	1.9842	4.5946	22.6938	1.8779	14.4938	4.4999
BCR3_5/3	0.8282	1.8557	4.2043	18.6272	1.9578	10.1061	4.1267
BCR3_6/1	0.7555	2.8588	4.2576	19.5338	2.0891	5.5202	4.3268
BCR3_6/2	0.6684	2.5852	4.3972	19.2798	2.1855	5.5253	4.5871
BCR3_6/3	0.5178	1.9539	3.8349	14.3330	2.1611	4.8803	4.1626
BCR3_7/1	0.6846	1.8926	3.8363	15.7412	1.9489	5.6012	4.3362
BCR3_7/2	0.8344	1.8446	4.2282	14.8338	1.9209	4.5677	3.9286
BCR3_7/3	0.8213	1.9872	4.7842	15.2569	2.1502	6.2929	3.9521
BCR3_8/1	0.6304	1.2157	3.0085	9.3409	1.4634	3.8130	3.0547
BCR3_8/2	0.5721	1.2948	3.2457	9.7626	1.4203	4.4248	3.3379
BCR3_8/3	0.5772	1.0546	2.8935	8.3785	1.5126	3.9760	3.0717
BCR3_9/1	0.6135	0.9770	2.7560	8.4897	1.4695	5.2197	3.6435
BCR3_9/2	0.4073	0.8693	2.6425	7.1551	1.5123	4.0582	2.9448

Table D-1. Total metal concentrations (Co, Cr, Cu, Mn, Ni, Pb and Zn) in 123 Soil tailings samples and CRM 050-025 (Cont.)

Samples	Metal concentration in Samples (mg/Kg)						
	Co	Cr	Cu	Mn	Ni	Pb	Zn
BCR3_9/3	0.5512	0.9091	2.8911	7.9688	1.2589	4.4737	2.8931
BCR3_10/1	0.3945	0.8883	2.6739	7.1334	1.5079	3.8903	2.9044
BCR3_10/2	0.4514	0.6788	2.4653	7.0540	1.7645	4.1123	2.8945
BCR3_10/3	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Res_1/1	8.0838	78.0727	50.5677	372.3242	9.6049	9.8845	61.2569
Res_1/2	7.0409	66.9221	46.8803	335.3210	7.2566	9.5129	55.0696
Res_1/3	7.8027	76.0265	49.9378	356.5041	8.5794	9.7700	59.2509
Res_2/1	7.6452	95.2889	41.7514	396.3364	13.3638	9.9444	66.1279
Res_2/2	7.8457	84.1496	44.5420	398.0429	8.6053	10.9696	65.1291
Res_2/3	7.5871	83.3820	41.4621	383.0155	7.8969	9.9791	62.1841
Res_3/1	6.7098	77.5059	37.2345	355.7632	7.5345	9.6830	66.1635
Res_3/2	6.6992	76.2916	50.0818	344.5242	7.4300	14.6868	79.7656
Res_3/3	8.1009	95.8507	60.2011	425.3943	8.8998	73.3606	152.0899
Res_4/1	9.6588	88.2026	63.2962	487.2389	8.1402	58.4923	135.0020
Res_4/2	10.7844	80.0984	63.0384	454.3898	8.9548	45.8740	95.9252
Res_4/3	10.7791	88.5973	68.5874	481.9191	10.5591	37.6110	111.0726
Res_5/1	9.5632	59.9240	50.6151	394.5794	8.3103	31.0535	85.8195
Res_5/2	9.4650	76.5355	53.5832	474.5501	8.7115	27.6518	93.6662
Res_5/3	7.8839	95.4854	56.5075	435.0434	8.9540	21.8962	80.9521
Res_6/1	9.0212	102.9528	60.5985	480.2021	11.0900	21.0295	102.7348
Res_6/2	9.2593	70.1276	49.5853	426.1563	10.3170	20.8224	89.1667
Res_6/3	8.9715	100.4711	58.7171	455.8108	9.2276	17.7778	80.9374
Res_7/1	10.3975	110.0319	58.8842	484.3893	10.8169	20.3447	90.4045
Res_7/2	9.3716	92.8930	59.8174	462.9218	9.8314	17.2956	82.2866
Res_7/3	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Res_8/1	7.8901	95.7324	50.4720	401.0677	10.0229	16.7561	72.5723
Res_8/2	8.2970	96.0920	51.6643	430.9377	9.3125	16.2114	76.5628
Res_8/3	8.1963	94.1542	54.6126	423.1818	8.5042	16.3636	78.0623
Res_9/1	10.7749	99.6239	65.4503	453.6223	13.6243	16.6637	81.8884
Res_9/2	10.2369	96.8871	59.7049	425.9475	12.2493	15.6603	72.5385
Res_9/3	11.0131	94.9395	63.6297	464.1099	10.2600	15.4425	77.1393
Res_10/1	8.4295	99.4452	54.1014	474.7276	9.9614	15.0198	76.6267
Res_10/2	8.5089	99.8004	55.6607	501.4371	9.0790	15.9980	73.7355
Res_10/3	8.8580	101.4773	54.9881	475.0942	9.1620	14.2861	74.3000
CRM1	7.1047	513.6521	16.8168	227.6899	27.6437	1699.9229	79.1516
CRM2	6.1510	484.4183	17.4865	264.0986	25.7743	1306.5678	74.5955
CRM3	6.3691	494.5055	19.8430	264.2857	27.9317	1343.9953	76.7053

*N/A means not available

Table D-2 Total heavy metal concentration in soil amendment ratio of triplicate control, 5%, 7% and 10% OM in 30 days at BCR1

Samples	Day	OM	Co1 mg/Kg	Cr1 mg/Kg	Cu1 mg/Kg	Mn1 mg/Kg	Ni1 mg/Kg	Pb1 mg/Kg	Zn1 mg/Kg
Ctrl1	1	-	2.8409	0.7149	6.8098	590.112	1.4488	1.4256	8.0299
Ctrl1	7	-	2.4415	0.5035	5.7532	523.802	1.5484	1.3978	7.0747
Ctrl1	15	-	3.1619	0.6169	6.7244	539.929	1.8954	0.9757	9.3128
Ctrl1	30	-	2.7998	0.5918	5.2331	634.893	1.9458	1.2282	8.4814
Ctrl2	1	-	2.9567	0.8219	6.8562	616.637	1.4594	1.6341	8.7237
Ctrl2	7	-	2.7414	0.5994	6.2887	600.518	1.8168	1.5862	8.0259
Ctrl2	15	-	2.8965	0.6006	5.8424	500.535	1.5116	1.1549	8.5151
Ctrl2	30	-	2.7282	0.4456	4.8446	621.065	1.8632	1.1148	9.0061
Ctrl3	1	-	2.7431	0.7418	6.3941	582.739	1.4223	1.2687	7.8270
Ctrl3	7	-	2.7429	0.5006	5.6594	592.984	1.5962	1.5452	8.3453
Ctrl3	15	-	3.1488	0.7890	6.4367	545.008	1.7718	1.3173	9.1002
Ctrl3	30	-	2.9008	0.8560	5.1513	659.159	2.2391	1.5374	9.2752
5%/1	1	5	2.3886	0.6680	0.8129	554.803	1.6463	0.7452	6.5287
5%/1	7	5	2.6063	0.5645	0.8427	612.811	2.2360	0.8708	6.8312
5%/1	15	5	3.1417	0.8055	0.8224	597.619	2.3425	1.0772	8.0399
5%/1	30	5	2.9207	0.4550	0.5316	738.049	2.9319	0.5091	8.0070
5%/2	1	5	2.8405	0.7937	1.0265	640.156	2.0891	0.6252	7.6507
5%/2	7	5	2.8280	0.7406	0.8419	664.394	2.3585	0.8883	7.3144
5%/2	15	5	3.1328	0.7992	0.8346	598.971	2.1063	1.0791	8.7218
5%/2	30	5	3.0591	0.4658	0.5383	751.862	2.7707	0.6480	8.0144
5%/3	1	5	2.8691	0.7309	1.1406	645.487	2.4422	0.8444	7.9738
5%/3	7	5	2.7739	0.7242	0.8772	650.541	2.0103	0.6954	7.6393
5%/3	15	5	3.0177	1.0903	0.7903	592.498	2.1431	0.6280	7.6116
5%/3	30	5	2.9151	0.5739	0.6108	743.999	2.5565	0.6120	7.8996
7%/1	1	7	2.5120	0.6974	0.6885	615.504	2.6629	0.6391	7.2990
7%/1	7	7	2.6544	0.6644	0.6101	655.627	2.3659	0.6598	6.5831
7%/1	15	7	2.6583	0.5780	0.5895	549.422	2.6030	0.2558	7.4787
7%/1	30	7	2.6902	0.5348	0.4675	713.690	2.9248	0.4949	8.9236
7%/2	1	7	2.4274	0.4527	0.9864	628.491	2.6046	0.3299	7.8504
7%/2	7	7	2.6635	0.6079	0.6154	652.046	2.2118	0.5227	6.7772
7%/2	15	7	2.9820	0.7896	0.6518	600.649	2.6787	0.4881	8.0066
7%/2	30	7	2.4878	0.5954	0.4900	687.399	2.9944	0.4232	7.3769
7%/3	1	7	2.5372	0.5312	0.5930	630.580	2.4489	0.3667	6.8268
7%/3	7	7	2.6891	0.6322	0.6377	679.068	2.2869	0.2493	7.1018
7%/3	15	7	2.8300	0.6753	0.6274	590.366	2.7377	0.8564	7.5376
7%/3	30	7	2.6310	0.4679	0.4235	711.911	2.7538	0.4622	7.2557

Table D-2 Total heavy metal concentration in soil amendment ratio of triplicate control, 5%, 7% and 10% OM in 30 days at BCR1 (Cont.)

Samples	Day	OM	Co1 mg/Kg	Cr1 mg/Kg	Cu1 mg/Kg	Mn1 mg/Kg	Ni1 mg/Kg	Pb1 mg/Kg	Zn1 mg/Kg
10%/1	1	10	2.2914	0.6692	0.5603	604.217	2.0645	0.2202	6.5438
10%/1	7	10	2.4701	0.4755	0.5251	653.356	2.2755	0.1702	6.9641
10%/1	15	10	2.7298	0.5943	0.5013	565.199	2.2707	1.0088	7.3755
10%/1	30	10	2.4417	0.4431	0.4610	707.448	3.0854	0.2962	6.8801
10%/2	1	10	2.4151	0.5447	0.5321	626.260	2.0648	0.5012	6.9233
10%/2	7	10	2.4672	0.3687	0.5256	637.834	2.2234	0.0913	6.5978
10%/2	15	10	2.5659	0.5234	0.5156	545.816	2.2470	1.3800	6.6228
10%/2	30	10	2.4138	0.4481	0.4157	709.268	2.8454	0.3059	6.6978
10%/3	1	10	2.4496	0.6133	0.5507	624.026	2.1626	0.4791	6.9748
10%/3	7	10	2.4821	0.5484	0.5015	650.594	2.3288	0.4399	6.2450
10%/3	15	10	2.6574	0.6036	0.5979	556.913	2.6636	0.8020	7.0031
10%/3	30	10	2.2074	0.4308	0.4320	685.658	2.7780	0.3152	6.9537

Table D-3 Total heavy metal concentration in soil amendment ratio of triplicate control, 5%, 7% and 10% OM in 30 days at BCR2

Samples	Day	OM	Co2 mg/Kg	Cr2 mg/Kg	Cu2 mg/Kg	Mn2 mg/Kg	Ni2 mg/Kg	Pb2 mg/Kg	Zn2 mg/Kg
Ctrl1	1	-	4.7747	14.7532	11.6128	764.434	4.3313	21.2727	8.9163
Ctrl1	7	-	3.8631	12.7440	10.4621	664.497	3.5862	19.7029	8.1270
Ctrl1	15	-	3.9032	16.3699	9.7292	651.071	4.3684	17.8604	9.8826
Ctrl1	30	-	3.8768	20.0202	12.2117	831.959	3.0664	29.9346	11.3818
Ctrl2	1	-	4.6132	14.7202	11.7066	760.197	4.2977	22.0590	8.7512
Ctrl2	7	-	4.3205	14.5240	12.0920	731.834	3.9504	22.9743	8.3440
Ctrl2	15	-	3.9136	16.2558	9.8602	643.120	4.3643	18.8561	9.7288
Ctrl2	30	-	4.1370	22.1560	12.8743	911.028	3.5902	32.9982	12.7808
Ctrl3	1	-	4.8209	15.1196	11.9401	771.350	4.3019	22.2045	9.1846
Ctrl3	7	-	4.2665	14.2065	12.1336	720.063	4.0749	21.5270	8.4300
Ctrl3	15	-	3.9025	15.9788	10.5503	607.665	4.1187	17.5464	9.5810
Ctrl3	30	-	3.8751	20.2331	11.8789	859.866	3.1892	31.9615	11.8357
5%/1	1	5	4.8256	11.8285	5.2089	738.487	4.8610	19.5650	10.0270
5%/1	7	5	4.2648	12.2566	4.4821	660.891	4.3493	19.8359	9.7127
5%/1	15	5	5.3747	14.8167	3.7749	648.397	5.3032	17.9185	12.3974
5%/1	30	5	5.3182	19.3923	5.6514	804.241	4.6180	33.3599	16.1397
5%/2	1	5	4.7865	11.9786	4.9703	756.217	4.8080	20.5996	9.9215
5%/2	7	5	4.0753	12.6660	4.4471	637.734	4.2065	20.5939	9.0019
5%/2	15	5	4.7017	13.1184	3.3859	580.211	5.0568	17.1952	11.2227
5%/2	30	5	4.7790	20.3715	6.1033	849.470	4.7597	33.7123	17.9408
5%/3	1	5	4.5848	11.7190	4.8654	716.591	4.5414	20.6243	9.7984
5%/3	7	5	4.1389	12.7653	4.7749	639.441	4.2220	20.5211	9.4728
5%/3	15	5	4.5763	14.1199	3.6529	608.097	4.7059	15.0378	10.7173
5%/3	30	5	4.4236	19.4409	5.9113	821.559	4.1453	34.3234	14.3649
7%/1	1	7	5.0999	11.4284	2.6345	714.218	5.0760	18.2726	10.9649
7%/1	7	7	4.2560	12.3232	2.5680	618.257	4.7807	17.8081	11.0792
7%/1	15	7	5.5736	12.5599	2.4337	654.971	5.3664	19.1332	13.0907
7%/1	30	7	5.8577	16.2502	4.2097	781.845	4.1714	23.6254	15.5008
7%/2	1	7	5.0515	12.0519	3.5267	710.417	5.3245	18.0103	12.8883
7%/2	7	7	4.6801	12.1102	2.8409	642.639	4.6835	19.2554	10.2758
7%/2	15	7	6.3904	13.6993	2.9786	708.992	5.7989	19.0598	14.2443
7%/2	30	7	6.2386	18.5650	4.0771	866.889	4.9807	29.8650	16.4097
7%/3	1	7	4.9226	11.3196	2.5281	697.077	5.3262	17.4212	10.9902
7%/3	7	7	4.5964	11.4968	2.5426	660.740	4.7390	18.1393	12.6602
7%/3	15	7	5.7155	13.8348	2.3063	659.796	5.7443	19.7333	13.2866
7%/3	30	7	5.8345	18.2682	4.2338	833.413	4.7157	29.1162	14.2700

Table D-3 Total heavy metal concentration in soil amendment ratio of triplicate control, 5%, 7% and 10% OM in 30 days at BCR2 (Cont.)

Samples	Day	OM	Co2 mg/Kg	Cr2 mg/Kg	Cu2 mg/Kg	Mn2 mg/Kg	Ni2 mg/Kg	Pb2 mg/Kg	Zn2 mg/Kg
10%/1	1	10	4.8977	10.6820	2.2678	689.432	5.3020	17.1254	11.1921
10%/1	7	10	4.8189	11.6016	2.0475	656.896	5.1406	17.8882	10.6418
10%/1	15	10	5.1978	13.0849	1.5420	583.022	5.3720	15.4364	11.9960
10%/1	30	10	4.8627	17.9724	2.7908	834.751	5.0105	29.9250	15.0504
10%/2	1	10	5.1552	10.6654	2.2901	719.227	5.3990	16.6972	11.2845
10%/2	7	10	4.8163	11.2377	2.0284	655.160	4.8430	17.5605	10.3812
10%/2	15	10	4.6872	11.8834	1.6078	537.383	5.0689	15.9124	11.4232
10%/2	30	10	4.5943	17.6772	2.8921	776.997	4.9910	28.7305	14.7217
10%/3	1	10	4.6058	9.6038	2.0440	651.014	4.9829	15.6831	10.4584
10%/3	7	10	4.5724	11.5236	2.0455	642.658	4.9246	17.6993	10.4607
10%/3	15	10	5.3554	12.9737	1.6408	590.682	5.8148	15.0786	12.1872
10%/3	30	10	3.6843	16.3267	2.9157	737.033	4.0967	21.5963	13.9141

Table D-4 Total heavy metal concentration in soil ratio of triplicate control, 5%, 7% and 10% OM in 30 days at BCR3

Samples	Day	OM	Co3 mg/Kg	Cr3 mg/Kg	Cu3 mg/Kg	Mn3 mg/Kg	Ni3 mg/Kg	Pb3 mg/Kg	Zn3 mg/Kg
Ctrl1	1	-	4.9690	3.7023	4.4793	16.0282	1.2186	3.3512	4.7741
Ctrl1	7	-	6.2829	2.9631	6.9449	17.0762	1.3815	1.8022	4.8344
Ctrl1	15	-	5.8262	3.6410	6.2698	24.7631	1.7753	1.8022	6.8671
Ctrl1	30	-	5.5467	1.9598	5.3811	10.8218	1.5589	2.1339	4.2619
Ctrl2	1	-	5.5615	2.4507	5.0393	16.0619	1.2701	3.6397	3.8790
Ctrl2	7	-	6.0422	2.8892	6.2916	16.1109	1.3158	2.6474	4.0276
Ctrl2	15	-	6.6794	3.3570	6.9402	25.8479	1.8486	2.6474	6.5091
Ctrl2	30	-	6.0389	3.3717	5.9300	11.6521	1.9063	2.0616	4.7406
Ctrl3	1	-	5.6463	3.3257	4.9690	16.7931	1.4888	3.2031	4.4083
Ctrl3	7	-	6.5092	3.1997	6.3880	16.4170	1.5744	2.6146	3.9816
Ctrl3	15	-	4.9165	4.1322	5.3007	22.5811	1.7179	2.6146	6.5527
Ctrl3	30	-	4.8344	2.6229	4.4015	9.9666	1.7359	2.5592	4.9119
5%/1	1	5	8.9249	4.2396	12.0922	27.6511	2.4052	4.9848	5.0924
5%/1	7	5	6.4441	3.5416	10.9446	24.3635	1.8447	4.3510	5.6859
5%/1	15	5	6.6008	4.2855	10.1116	46.3706	2.0542	4.3510	4.9776
5%/1	30	5	4.0810	3.2135	8.9281	12.8394	1.7675	2.7932	4.9218
5%/2	1	5	7.7360	4.5011	11.4586	26.8831	1.8988	5.4926	5.0334
5%/2	7	5	6.1061	4.5425	10.3614	24.1912	1.9675	3.9899	5.1603
5%/2	15	5	7.3576	4.0718	10.5251	46.4478	1.8716	3.9899	8.5348
5%/2	30	5	9.5643	4.6606	15.9095	19.2429	1.8015	3.1836	6.3923
5%/3	1	5	7.2440	4.0092	10.6890	24.6405	1.7447	5.3401	4.6386
5%/3	7	5	8.2905	6.0727	13.6752	28.6091	2.2725	4.6653	5.9923
5%/3	15	5	9.2089	5.5773	11.6862	53.5508	1.9541	4.6653	11.4168
5%/3	30	5	6.7834	3.7154	12.1187	15.9696	1.8379	2.7716	5.4982
7%/1	1	7	10.1344	5.5706	16.0008	37.4545	2.5510	5.7803	5.5200
7%/1	7	7	6.5155	6.8296	11.6788	25.4522	1.9944	4.9418	5.0739
7%/1	15	7	8.6375	5.5348	12.8605	65.7992	1.7248	4.9418	8.8446
7%/1	30	7	6.9699	4.7838	13.4342	19.0318	1.9142	4.1354	6.1397
7%/2	1	7	7.3319	5.5687	13.8183	34.0382	2.7106	5.4357	6.6670
7%/2	7	7	9.9030	7.0616	16.9484	34.7965	2.7829	4.2485	7.1943
7%/2	15	7	11.8174	7.0538	16.0114	27.8102	1.7898	4.2485	9.4647
7%/2	30	7	8.0977	7.2246	14.9073	20.8240	2.0403	4.4555	6.0316
7%/3	1	7	6.9954	5.3206	12.1512	29.7684	2.0977	5.2136	5.7524
7%/3	7	7	8.8171	5.4791	15.0447	33.7189	2.8123	4.2426	6.7714
7%/3	15	7	7.5853	4.8248	10.9456	56.9862	1.7308	4.2426	7.6664
7%/3	30	7	6.4838	4.9128	11.5422	16.5223	1.8282	3.5265	5.4548

Table D-4 Total heavy metal concentration in soil ratio of triplicate control, 5%, 7% and 10% OM in 30 days at BCR3 (Cont.)

Samples	Day	OM	Co3 mg/Kg	Cr3 mg/Kg	Cu3 mg/Kg	Mn3 mg/Kg	Ni3 mg/Kg	Pb3 mg/Kg	Zn3 mg/Kg
10%/1	1	10	7.8326	6.8771	13.5730	31.6987	2.4277	6.3410	6.3432
10%/1	7	10	9.4015	9.4795	16.6409	37.1537	2.8562	5.9922	7.3660
10%/1	15	10	8.3294	12.9394	11.2594	68.3777	1.8423	5.9922	10.5733
10%/1	30	10	8.1505	6.5461	16.3647	23.7865	1.7950	3.8148	7.1092
10%/2	1	10	6.8467	7.4732	12.7214	31.8575	2.3138	6.0198	6.6191
10%/2	7	10	6.1842	4.7425	11.2933	29.9813	2.2198	5.5349	6.0942
10%/2	15	10	9.1361	5.7004	11.8756	71.2560	2.0019	5.5349	9.1800
10%/2	30	10	6.3299	5.0632	12.2123	19.8007	1.9186	4.4452	7.8930
10%/3	1	10	6.2186	6.7466	12.7428	30.5838	2.1094	7.6414	5.9007
10%/3	7	10	9.5883	7.3411	16.6864	38.6673	3.2859	5.8638	7.6965
10%/3	15	10	13.9184	8.0718	18.1092	34.6256	2.0221	5.8638	6.9144
10%/3	30	10	5.0540	5.7330	11.4944	18.8922	1.8348	3.3168	6.1323

Table D-5 Total heavy metal concentration in soil amendment ratio of triplicate control, 5%, 7% and 10% OM in 30 days at residual fraction

Samples	Day	OM	Co4 mg/Kg	Cr4 mg/Kg	Cu4 mg/Kg	Mn4 mg/Kg	Ni4 mg/Kg	Pb4 mg/Kg	Zn4 mg/Kg
Ctrl1	1	-	6.8204	73.1948	57.8648	424.601	7.9338	12.5837	67.8977
Ctrl1	7	-	7.3165	86.0893	45.3055	418.431	11.271	11.0843	69.6655
Ctrl1	15	-	8.262	96.2322	35.5551	339.958	9.8979	61.813	65.2458
Ctrl1	30	-	8.3755	109.824	38.0174	417.486	7.2862	11.7268	71.7803
Ctrl2	1	-	7.9509	93.703	58.8574	424.848	9.9951	11.6696	71.0383
Ctrl2	7	-	8.2601	93.9697	47.5393	434.810	11.7081	10.0836	73.0768
Ctrl2	15	-	8.2604	99.6436	37.0432	347.446	9.7049	61.794	67.9875
Ctrl2	30	-	8.5433	116.145	35.8513	425.262	9.1086	11.8491	70.8068
Ctrl3	1	-	8.0857	92.0266	59.1961	441.232	9.1384	11.6719	67.5133
Ctrl3	7	-	7.641	89.53	46.2495	424.547	10.1941	10.9854	69.9951
Ctrl3	15	-	8.3368	103.322	37.0106	351.012	12.2239	11.3287	70.6692
Ctrl3	30	-	8.3032	109.955	36.3473	420.656	7.53	12.0617	70.8962
5%/1	1	5	6.5713	79.5969	55.8896	428.392	8.12	14.2127	71.3564
5%/1	7	5	6.9239	90.7924	43.1571	417.834	10.1066	11.2084	79.584
5%/1	15	5	7.5269	105.424	34.7924	323.832	14.3915	70.87	68.2893
5%/1	30	5	7.8096	98.3812	32.9522	397.315	6.7754	11.7946	65.6433
5%/2	1	5	6.5606	88.7376	51.2247	396.559	9.0583	12.9782	67.5784
5%/2	7	5	6.8844	99.16	43.579	425.902	11.347	10.9086	67.1623
5%/2	15	5	7.1448	102.381	34.4937	314.751	14.9113	64.9595	67.1125
5%/2	30	5	8.1452	110.883	32.5084	408.935	7.9792	11.1239	63.529
5%/3	1	5	6.0856	73.9356	54.7526	429.096	7.7428	12.7605	67.6195
5%/3	7	5	7.1267	92.8796	43.0642	426.575	10.0038	11.4054	70.0079
5%/3	15	5	7.4442	102.366	31.8967	308.966	15.9321	61.9315	66.1501
5%/3	30	5	8.5629	108.388	33.3919	420.057	7.8719	11.1132	70.5105
7%/1	1	7	5.513	75.7836	69.5987	361.041	7.5825	48.3595	113.94
7%/1	7	7	6.4468	105.846	41.9218	422.678	10.9916	10.8554	73.7613
7%/1	15	7	5.6702	92.4394	27.8549	247.801	9.8466	57.0358	48.859
7%/1	30	7	6.5266	95.0351	28.3808	340.687	8.2998	9.5352	52.4416
7%/2	1	7	6.0526	79.0531	58.3349	410.553	8.6652	17.0669	65.6198
7%/2	7	7	7.3543	96.3441	45.994	436.456	10.533	12.5471	78.7981
7%/2	15	7	6.4295	97.4037	29.7659	266.405	10.2727	44.7538	52.5944
7%/2	30	7	8.831	120.2	34.8199	424.940	9.8243	12.3696	67.0606
7%/3	1	7	6.1302	84.2515	51.8182	386.881	9.3212	14.3933	66.0283
7%/3	7	7	6.6306	90.0387	42.7711	408.082	10.1519	11.268	71.3451
7%/3	15	7	6.9172	95.2412	31.3357	282.391	10.9085	64.363	57.085
7%/3	30	7	8.2233	112.093	33.3389	417.566	7.8921	12.3081	72.9913

Table D-5 Total heavy metal concentration in soil amendment ratio of triplicate control, 5%, 7% and 10% OM in 30 days at residual fraction (Cont.)

Samples	Day	OM	Co4 mg/Kg	Cr4 mg/Kg	Cu4 mg/Kg	Mn4 mg/Kg	Ni4 mg/Kg	Pb4 mg/Kg	Zn4 mg/Kg
10%/1	1	10	6.9587	87.3637	54.337	411.503	9.9168	13.3167	64.5105
10%/1	7	10	6.5538	104.716	42.8166	418.697	10.649	11.2894	65.2493
10%/1	15	10	7.176	103.49	31.6788	304.011	11.3367	78.3966	62.531
10%/1	30	10	8.826	101.514	33.8721	400.300	10.2056	12.9156	67.6357
10%/2	1	10	6.4303	80.1746	52.257	392.415	9.0324	12.044	64.6854
10%/2	7	10	6.6989	91.8655	43.4293	421.654	10.2216	11.6254	66.981
10%/2	15	10	7.219	97.2708	32.9362	309.135	11.7258	53.5785	61.979
10%/2	30	10	8.0128	105.325	33.1353	397.182	8.437	11.9616	71.277
10%/3	1	10	6.5459	79.7628	54.2093	418.766	9.0871	13.3609	64.7824
10%/3	7	10	7.3031	95.2647	42.9897	424.982	12.0813	11.6933	66.3951
10%/3	15	10	7.3238	97.4951	33.1678	295.706	9.9926	77.147	60.2057
10%/3	30	10	8.5113	110.968	35.5052	413.203	7.9955	12.4299	68.5691

Appendix E

Table E-1.1a Day 1; BCR1 ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co1	Between Groups	.385	3	.128	5.445	.025
	Within Groups	.189	8	.024		
	Total	.574	11			
Cr1	Between Groups	.082	3	.027	4.119	.049
	Within Groups	.053	8	.007		
	Total	.135	11			
Cu1	Between Groups	79.180	3	26.393	783.586	.000
	Within Groups	.269	8	.034		
	Total	79.450	11			
Mn1	Between Groups	1319.087	3	439.696	.563	.654
	Within Groups	6242.610	8	780.326		
	Total	7561.696	11			
Ni1	Between Groups	1.928	3	.643	14.701	.001
	Within Groups	.350	8	.044		
	Total	2.277	11			
Pb1	Between Groups	2.086	3	.695	28.206	.000
	Within Groups	.197	8	.025		
	Total	2.283	11			
Zn1	Between Groups	2.926	3	.975	3.502	.069
	Within Groups	2.228	8	.279		
	Total	5.155	11			

Table E-1.1b Day 1; Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co1	.00	.05	.1475000	.1253748	.273
		.07	.3547000*	.1253748	.022
		.10	.4615333*	.1253748	.006
	.05	.00	-.1475000	.1253748	.273
		.07	.2072000	.1253748	.137
		.10	.3140333*	.1253748	.037
	.07	.00	-.3547000*	.1253748	.022
		.05	-.2072000	.1253748	.137
		.10	.1068333	.1253748	.419
	.10	.00	-.4615333*	.1253748	.006
		.05	-.3140333*	.1253748	.037
		.07	-.1068333	.1253748	.419
Cr1	.00	.05	.0286667	.0665172	.678
		.07	.1991000*	.0665172	.017
		.10	.1504667	.0665172	.054
	.05	.00	-.0286667	.0665172	.678
		.07	.1704333*	.0665172	.034
		.10	.1218000	.0665172	.104
	.07	.00	-.1991000*	.0665172	.017
		.05	-.1704333*	.0665172	.034
		.10	-.0486333	.0665172	.486
	.10	.00	-.1504667	.0665172	.054
		.05	-.1218000	.0665172	.104
		.07	.0486333	.0665172	.486
Cu1	.00	.05	5.6933667*	.1498507	.000
		.07	5.9307333*	.1498507	.000
		.10	6.1390000*	.1498507	.000
	.05	.00	-5.6933667*	.1498507	.000
		.07	.2373667	.1498507	.152
		.10	.4456333*	.1498507	.018
	.07	.00	-5.9307333*	.1498507	.000
		.05	-.2373667	.1498507	.152
		.10	.2082667	.1498507	.202
	.10	.00	-6.1390000*	.1498507	.000
		.05	-.4456333*	.1498507	.018
		.07	-.2082667	.1498507	.202
Mn1	.00	.05	-16.9860667	22.8082761	.478
		.07	-28.3626667	22.8082761	.249
		.10	-21.6717333	22.8082761	.370
	.05	.00	16.9860667	22.8082761	.478
		.07	-11.3766000	22.8082761	.631
		.10	-4.6856667	22.8082761	.842
	.07	.00	28.3626667	22.8082761	.249
		.05	11.3766000	22.8082761	.631
		.10	6.6909333	22.8082761	.777
	.10	.00	21.6717333	22.8082761	.370
		.05	4.6856667	22.8082761	.842
		.07	-6.6909333	22.8082761	.777

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni1	.00	.05	-.6157000*	.1707032	.007
		.07	-1.1286333*	.1707032	.000
		.10	-.6538000*	.1707032	.005
	.05	.00	.6157000*	.1707032	.007
		.07	-.5129333*	.1707032	.017
		.10	-.0381000	.1707032	.829
	.07	.00	1.1286333*	.1707032	.000
		.05	.5129333*	.1707032	.017
		.10	.4748333*	.1707032	.024
	.10	.00	.6538000*	.1707032	.005
		.05	.0381000	.1707032	.829
		.07	-.4748333*	.1707032	.024
Pb1	.00	.05	.7045333*	.1281856	.001
		.07	.9975667*	.1281856	.000
		.10	1.0426333*	.1281856	.000
	.05	.00	-.7045333*	.1281856	.001
		.07	.2930333	.1281856	.052
		.10	.3381000*	.1281856	.030
	.07	.00	-.9975667*	.1281856	.000
		.05	-.2930333	.1281856	.052
		.10	.0450667	.1281856	.734
	.10	.00	-1.0426333*	.1281856	.000
		.05	-.3381000*	.1281856	.030
		.07	-.0450667	.1281856	.734
Zn1	.00	.05	.8091333	.4309355	.097
		.07	.8681333	.4309355	.079
		.10	1.3795667*	.4309355	.013
	.05	.00	-.8091333	.4309355	.097
		.07	.0590000	.4309355	.894
		.10	.5704333	.4309355	.222
	.07	.00	-.8681333	.4309355	.079
		.05	-.0590000	.4309355	.894
		.10	.5114333	.4309355	.269
	.10	.00	-1.3795667*	.4309355	.013
		.05	-.5704333	.4309355	.222
		.07	-.5114333	.4309355	.269

Table E-1.2a Day 1; BCR2 ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co2	Between Groups	.176	3	.059	2.079	.181
	Within Groups	.225	8	.028		
	Total	.401	11			
Cr2	Between Groups	33.373	3	11.124	73.684	.000
	Within Groups	1.208	8	.151		
	Total	34.581	11			
Cu2	Between Groups	170.992	3	56.997	602.067	.000
	Within Groups	.757	8	.095		
	Total	171.749	11			
Mn2	Between Groups	10687.154	3	3562.385	8.501	.007
	Within Groups	3352.356	8	419.045		
	Total	14039.511	11			
Ni2	Between Groups	1.792	3	.597	24.433	.000
	Within Groups	.196	8	.024		
	Total	1.988	11			
Pb2	Between Groups	51.220	3	17.073	50.380	.000
	Within Groups	2.711	8	.339		
	Total	53.932	11			
Zn2	Between Groups	12.419	3	4.140	11.166	.003
	Within Groups	2.966	8	.371		
	Total	15.384	11			

Table E-1.2b Day 1; BCR2 Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co2	.00	.05	.0039667	.1369608	.978
		.07	-.2884000	.1369608	.068
		.10	-.1499667	.1369608	.305
	.05	.00	-.0039667	.1369608	.978
		.07	-.2923667	.1369608	.065
		.10	-.1539333	.1369608	.294
	.07	.00	.2884000	.1369608	.068
		.05	.2923667	.1369608	.065
		.10	.1384333	.1369608	.342
	.10	.00	.1499667	.1369608	.305
		.05	.1539333	.1369608	.294
		.07	-.1384333	.1369608	.342
Cr2	.00	.05	3.0223000*	.3172542	.000
		.07	3.2643667*	.3172542	.000
		.10	4.5472667*	.3172542	.000
	.05	.00	-3.0223000*	.3172542	.000
		.07	.2420667	.3172542	.467
		.10	1.5249667*	.3172542	.001
	.07	.00	-3.2643667*	.3172542	.000
		.05	-.2420667	.3172542	.467
		.10	1.2829000*	.3172542	.004
	.10	.00	-4.5472667*	.3172542	.000
		.05	-1.5249667*	.3172542	.001
		.07	-1.2829000*	.3172542	.004
Cu2	.00	.05	6.7383000*	.2512228	.000
		.07	8.8567333*	.2512228	.000
		.10	9.5525333*	.2512228	.000
	.05	.00	-6.7383000*	.2512228	.000
		.07	2.1184333*	.2512228	.000
		.10	2.8142333*	.2512228	.000
	.07	.00	-8.8567333*	.2512228	.000
		.05	-2.1184333*	.2512228	.000
		.10	.6958000*	.2512228	.024
	.10	.00	-9.5525333*	.2512228	.000
		.05	-2.8142333*	.2512228	.000
		.07	-.6958000*	.2512228	.024
Mn2	.00	.05	28.2284000	16.7141561	.130
		.07	58.0893667*	16.7141561	.008
		.10	78.7693000*	16.7141561	.002
	.05	.00	-28.2284000	16.7141561	.130
		.07	29.8609667	16.7141561	.112
		.10	50.5409000*	16.7141561	.016
	.07	.00	-58.0893667*	16.7141561	.008
		.05	-29.8609667	16.7141561	.112
		.10	20.6799333	16.7141561	.251
	.10	.00	-78.7693000*	16.7141561	.002
		.05	-50.5409000*	16.7141561	.016
		.07	-20.6799333	16.7141561	.251

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni2	.00	.05	-.4265000*	.1276680	.010
		.07	-.9319333*	.1276680	.000
		.10	-.9176667*	.1276680	.000
	.05	.00	.4265000*	.1276680	.010
		.07	-.5054333*	.1276680	.004
		.10	-.4911667*	.1276680	.005
	.07	.00	.9319333*	.1276680	.000
		.05	.5054333*	.1276680	.004
		.10	.0142667	.1276680	.914
	.10	.00	.9176667*	.1276680	.000
		.05	.4911667*	.1276680	.005
		.07	-.0142667	.1276680	.914
Pb2	.00	.05	1.5824333*	.4753221	.010
		.07	3.9440333*	.4753221	.000
		.10	5.3435000*	.4753221	.000
	.05	.00	-1.5824333*	.4753221	.010
		.07	2.3616000*	.4753221	.001
		.10	3.7610667*	.4753221	.000
	.07	.00	-3.9440333*	.4753221	.000
		.05	-2.3616000*	.4753221	.001
		.10	1.3994667*	.4753221	.019
	.10	.00	-5.3435000*	.4753221	.000
		.05	-3.7610667*	.4753221	.000
		.07	-1.3994667*	.4753221	.019
Zn2	.00	.05	-.9649333	.4971532	.088
		.07	-2.6637667*	.4971532	.001
		.10	-2.0276333*	.4971532	.004
	.05	.00	.9649333	.4971532	.088
		.07	-1.6988333*	.4971532	.009
		.10	-1.0627000	.4971532	.065
	.07	.00	2.6637667*	.4971532	.001
		.05	1.6988333*	.4971532	.009
		.10	.6361333	.4971532	.237
	.10	.00	2.0276333*	.4971532	.004
		.05	1.0627000	.4971532	.065
		.07	-.6361333	.4971532	.237

Table E-1.3a Day 1; BCR3 ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co3	Between Groups	14.392	3	4.797	4.250	.045
	Within Groups	9.030	8	1.129		
	Total	23.422	11			
Cr3	Between Groups	24.947	3	8.316	51.679	.000
	Within Groups	1.287	8	.161		
	Total	26.234	11			
Cu3	Between Groups	153.292	3	51.097	44.924	.000
	Within Groups	9.099	8	1.137		
	Total	162.392	11			
Mn3	Between Groups	539.306	3	179.769	40.070	.000
	Within Groups	35.891	8	4.486		
	Total	575.197	11			
Ni3	Between Groups	2.217	3	.739	11.066	.003
	Within Groups	.534	8	.067		
	Total	2.751	11			
Pb3	Between Groups	16.446	3	5.482	23.427	.000
	Within Groups	1.872	8	.234		
	Total	18.318	11			
Zn3	Between Groups	7.340	3	2.447	12.837	.002
	Within Groups	1.525	8	.191		
	Total	8.865	11			

Table E-1.3b Day 1; BCR3 Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co3	.00	.05	-2.5760333*	.8674679	.018
		.07	-2.7616333*	.8674679	.013
		.10	-1.5737000	.8674679	.107
	.05	.00	2.5760333*	.8674679	.018
		.07	-.1856000	.8674679	.836
		.10	1.0023333	.8674679	.281
	.07	.00	2.7616333*	.8674679	.013
		.05	.1856000	.8674679	.836
		.10	1.1879333	.8674679	.208
	.10	.00	1.5737000	.8674679	.107
		.05	-1.0023333	.8674679	.281
		.07	-1.1879333	.8674679	.208
Cr3	.00	.05	-1.0904000*	.3275226	.010
		.07	-2.3270667*	.3275226	.000
		.10	-3.8727333*	.3275226	.000
	.05	.00	1.0904000*	.3275226	.010
		.07	-1.2366667*	.3275226	.005
		.10	-2.7823333*	.3275226	.000
	.07	.00	2.3270667*	.3275226	.000
		.05	1.2366667*	.3275226	.005
		.10	-1.5456667*	.3275226	.002
	.10	.00	3.8727333*	.3275226	.000
		.05	2.7823333*	.3275226	.000
		.07	1.5456667*	.3275226	.002
Cu3	.00	.05	-6.5840667*	.8707901	.000
		.07	-9.1609000*	.8707901	.000
		.10	-8.1832000*	.8707901	.000
	.05	.00	6.5840667*	.8707901	.000
		.07	-2.5768333*	.8707901	.018
		.10	-1.5991333	.8707901	.104
	.07	.00	9.1609000*	.8707901	.000
		.05	2.5768333*	.8707901	.018
		.10	.9777000	.8707901	.294
	.10	.00	8.1832000*	.8707901	.000
		.05	1.5991333	.8707901	.104
		.07	-.9777000	.8707901	.294
Mn3	.00	.05	-10.0971667*	1.7294236	.000
		.07	-17.4593000*	1.7294236	.000
		.10	-15.0856000*	1.7294236	.000
	.05	.00	10.0971667*	1.7294236	.000
		.07	-7.3621333*	1.7294236	.003
		.10	-4.9884333*	1.7294236	.020
	.07	.00	17.4593000*	1.7294236	.000
		.05	7.3621333*	1.7294236	.003
		.10	2.3737000	1.7294236	.207
	.10	.00	15.0856000*	1.7294236	.000
		.05	4.9884333*	1.7294236	.020
		.07	-2.3737000	1.7294236	.207

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni3	.00	.05	-.6904000*	.2109901	.011
		.07	-1.1272667*	.2109901	.001
		.10	-.9578000*	.2109901	.002
	.05	.00	.6904000*	.2109901	.011
		.07	-.4368667	.2109901	.072
		.10	-.2674000	.2109901	.241
	.07	.00	1.1272667*	.2109901	.001
		.05	.4368667	.2109901	.072
		.10	.1694667	.2109901	.445
	.10	.00	.9578000*	.2109901	.002
		.05	.2674000	.2109901	.241
		.07	-.1694667	.2109901	.445
Pb3	.00	.05	-1.8745000*	.3949741	.001
		.07	-2.0785333*	.3949741	.001
		.10	-3.2694000*	.3949741	.000
	.05	.00	1.8745000*	.3949741	.001
		.07	-.2040333	.3949741	.619
		.10	-1.3949000*	.3949741	.008
	.07	.00	2.0785333*	.3949741	.001
		.05	.2040333	.3949741	.619
		.10	-1.1908667*	.3949741	.017
	.10	.00	3.2694000*	.3949741	.000
		.05	1.3949000*	.3949741	.008
		.07	1.1908667*	.3949741	.017
Zn3	.00	.05	-.5676667	.3564716	.150
		.07	-1.6260000*	.3564716	.002
		.10	-1.9338667*	.3564716	.001
	.05	.00	.5676667	.3564716	.150
		.07	-1.0583333*	.3564716	.018
		.10	-1.3662000*	.3564716	.005
	.07	.00	1.6260000*	.3564716	.002
		.05	1.0583333*	.3564716	.018
		.10	-.3078667	.3564716	.413
	.10	.00	1.9338667*	.3564716	.001
		.05	1.3662000*	.3564716	.005
		.07	.3078667	.3564716	.413

Table E-1.4a Day 1; RES ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co4	Between Groups	4.689	3	1.563	8.336	.008
	Within Groups	1.500	8	.187		
	Total	6.189	11			
Cr4	Between Groups	75.736	3	25.245	.455	.721
	Within Groups	443.927	8	55.491		
	Total	519.662	11			
Cu4	Between Groups	93.387	3	31.129	1.404	.311
	Within Groups	177.338	8	22.167		
	Total	270.725	11			
Mn4	Between Groups	3140.371	3	1046.790	3.391	.074
	Within Groups	2469.573	8	308.697		
	Total	5609.944	11			
Ni4	Between Groups	2.000	3	.667	1.047	.423
	Within Groups	5.095	8	.637		
	Total	7.096	11			
Pb4	Between Groups	435.900	3	145.300	1.623	.259
	Within Groups	716.260	8	89.533		
	Total	1152.161	11			
Zn4	Between Groups	502.718	3	167.573	.859	.500
	Within Groups	1560.411	8	195.051		
	Total	2063.129	11			

Table E-1.4b Day 1; RES Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co4	.00	.05	1.2131667*	.3535506	.009
		.07	1.7204000*	.3535506	.001
		.10	.9740333*	.3535506	.025
	.05	.00	-1.2131667*	.3535506	.009
		.07	.5072333	.3535506	.189
		.10	-.2391333	.3535506	.518
	.07	.00	-1.7204000*	.3535506	.001
		.05	-.5072333	.3535506	.189
		.10	-.7463667	.3535506	.068
	.10	.00	-.9740333*	.3535506	.025
		.05	.2391333	.3535506	.518
		.07	.7463667	.3535506	.068
Cr4	.00	.05	5.5514333	6.0822600	.388
		.07	6.6120667	6.0822600	.309
		.10	3.8744333	6.0822600	.542
	.05	.00	-5.5514333	6.0822600	.388
		.07	1.0606333	6.0822600	.866
		.10	-1.6770000	6.0822600	.790
	.07	.00	-6.6120667	6.0822600	.309
		.05	-1.0606333	6.0822600	.866
		.10	-2.7376333	6.0822600	.665
	.10	.00	-3.8744333	6.0822600	.542
		.05	1.6770000	6.0822600	.790
		.07	2.7376333	6.0822600	.665
Cu4	.00	.05	4.6838000	3.8442360	.258
		.07	-1.2778333	3.8442360	.748
		.10	5.0383333	3.8442360	.226
	.05	.00	-4.6838000	3.8442360	.258
		.07	-5.9616333	3.8442360	.160
		.10	.3545333	3.8442360	.929
	.07	.00	1.2778333	3.8442360	.748
		.05	5.9616333	3.8442360	.160
		.10	6.3161667	3.8442360	.139
	.10	.00	-5.0383333	3.8442360	.226
		.05	-.3545333	3.8442360	.929
		.07	-6.3161667	3.8442360	.139
Mn4	.00	.05	12.2110333	14.3456520	.419
		.07	44.0686000*	14.3456520	.015
		.10	22.6652000	14.3456520	.153
	.05	.00	-12.2110333	14.3456520	.419
		.07	31.8575667	14.3456520	.057
		.10	10.4541667	14.3456520	.487
	.07	.00	-44.0686000*	14.3456520	.015
		.05	-31.8575667	14.3456520	.057
		.10	-21.4034000	14.3456520	.174
	.10	.00	-22.6652000	14.3456520	.153
		.05	-10.4541667	14.3456520	.487
		.07	21.4034000	14.3456520	.174

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni4	.00	.05	.7154000	.6516302	.304
		.07	.4994667	.6516302	.465
		.10	-.3230000	.6516302	.633
	.05	.00	-.7154000	.6516302	.304
		.07	-.2159333	.6516302	.749
		.10	-1.0384000	.6516302	.150
	.07	.00	-.4994667	.6516302	.465
		.05	.2159333	.6516302	.749
		.10	-.8224667	.6516302	.242
	.10	.00	.3230000	.6516302	.633
		.05	1.0384000	.6516302	.150
		.07	.8224667	.6516302	.242
Pb4	.00	.05	-1.3420667	7.7258241	.866
		.07	-14.6315000	7.7258241	.095
		.10	-.9321333	7.7258241	.907
	.05	.00	1.3420667	7.7258241	.866
		.07	-13.2894333	7.7258241	.124
		.10	.4099333	7.7258241	.959
	.07	.00	14.6315000	7.7258241	.095
		.05	13.2894333	7.7258241	.124
		.10	13.6993667	7.7258241	.114
	.10	.00	.9321333	7.7258241	.907
		.05	-.4099333	7.7258241	.959
		.07	-13.6993667	7.7258241	.114
Zn4	.00	.05	-.0350000	11.4032567	.998
		.07	-13.0461000	11.4032567	.286
		.10	4.1570000	11.4032567	.725
	.05	.00	.0350000	11.4032567	.998
		.07	-13.0111000	11.4032567	.287
		.10	4.1920000	11.4032567	.723
	.07	.00	13.0461000	11.4032567	.286
		.05	13.0111000	11.4032567	.287
		.10	17.2031000	11.4032567	.170
	.10	.00	-4.1570000	11.4032567	.725
		.05	-4.1920000	11.4032567	.723
		.07	-17.2031000	11.4032567	.170

Table E-1.5a Day 7; BCR1 ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co1	Between Groups	.113	3	.038	3.420	.073
	Within Groups	.088	8	.011		
	Total	.200	11			
Cr1	Between Groups	.083	3	.028	5.142	.029
	Within Groups	.043	8	.005		
	Total	.126	11			
Cu1	Between Groups	61.871	3	20.624	710.708	.000
	Within Groups	.232	8	.029		
	Total	62.103	11			
Mn1	Between Groups	14414.392	3	4804.797	6.902	.013
	Within Groups	5569.451	8	696.181		
	Total	19983.843	11			
Ni1	Between Groups	.827	3	.276	18.254	.001
	Within Groups	.121	8	.015		
	Total	.948	11			
Pb1	Between Groups	2.767	3	.922	37.535	.000
	Within Groups	.197	8	.025		
	Total	2.963	11			
Zn1	Between Groups	2.583	3	.861	4.304	.044
	Within Groups	1.600	8	.200		
	Total	4.183	11			

Table E-1.5b Day 7; BCR1 Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co1	.00	.05	-.0941333	.0855161	.303
		.07	-.0270667	.0855161	.760
		.10	.1688000	.0855161	.084
	.05	.00	.0941333	.0855161	.303
		.07	.0670667	.0855161	.455
		.10	.2629333*	.0855161	.015
	.07	.00	.0270667	.0855161	.760
		.05	-.0670667	.0855161	.455
		.10	.1958667	.0855161	.051
	.10	.00	-.1688000	.0855161	.084
		.05	-.2629333*	.0855161	.015
		.07	-.1958667	.0855161	.051
Cr1	.00	.05	-.1419333*	.0599961	.046
		.07	-.1003333	.0599961	.133
		.10	.0703000	.0599961	.275
	.05	.00	.1419333*	.0599961	.046
		.07	.0416000	.0599961	.508
		.10	.2122333*	.0599961	.008
	.07	.00	.1003333	.0599961	.133
		.05	-.0416000	.0599961	.508
		.10	.1706333*	.0599961	.022
	.10	.00	-.0703000	.0599961	.275
		.05	-.2122333*	.0599961	.008
		.07	-.1706333*	.0599961	.022
Cu1	.00	.05	5.0465000*	.1390882	.000
		.07	5.2793667*	.1390882	.000
		.10	5.3830333*	.1390882	.000
	.05	.00	-5.0465000*	.1390882	.000
		.07	.2328667	.1390882	.133
		.10	.3365333*	.1390882	.042
	.07	.00	-5.2793667*	.1390882	.000
		.05	-.2328667	.1390882	.133
		.10	.1036667	.1390882	.477
	.10	.00	-5.3830333*	.1390882	.000
		.05	-.3365333*	.1390882	.042
		.07	-.1036667	.1390882	.477
Mn1	.00	.05	-70.1472667*	21.5434660	.012
		.07	-89.8126000*	21.5434660	.003
		.10	-74.8268000*	21.5434660	.008
	.05	.00	70.1472667*	21.5434660	.012
		.07	-19.6653333	21.5434660	.388
		.10	-4.6795333	21.5434660	.833
	.07	.00	89.8126000*	21.5434660	.003
		.05	19.6653333	21.5434660	.388
		.10	14.9858000	21.5434660	.506
	.10	.00	74.8268000*	21.5434660	.008
		.05	4.6795333	21.5434660	.833
		.07	-14.9858000	21.5434660	.506

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni1	.00	.05	-.5478000*	.1003425	.001
		.07	-.6344000*	.1003425	.000
		.10	-.6221000*	.1003425	.000
	.05	.00	.5478000*	.1003425	.001
		.07	-.0866000	.1003425	.413
		.10	-.0743000	.1003425	.480
	.07	.00	.6344000*	.1003425	.000
		.05	.0866000	.1003425	.413
		.10	.0123000	.1003425	.905
	.10	.00	.6221000*	.1003425	.000
		.05	.0743000	.1003425	.480
		.07	-.0123000	.1003425	.905
Pb1	.00	.05	.6915667*	.1279891	.001
		.07	1.0324667*	.1279891	.000
		.10	1.2759333*	.1279891	.000
	.05	.00	-.6915667*	.1279891	.001
		.07	.3409000*	.1279891	.029
		.10	.5843667*	.1279891	.002
	.07	.00	-1.0324667*	.1279891	.000
		.05	-.3409000*	.1279891	.029
		.10	.2434667	.1279891	.094
	.10	.00	-1.2759333*	.1279891	.000
		.05	-.5843667*	.1279891	.002
		.07	-.2434667	.1279891	.094
Zn1	.00	.05	.5536667	.3651913	.168
		.07	.9946000*	.3651913	.026
		.10	1.2130000*	.3651913	.011
	.05	.00	-.5536667	.3651913	.168
		.07	.4409333	.3651913	.262
		.10	.6593333	.3651913	.109
	.07	.00	-.9946000*	.3651913	.026
		.05	-.4409333	.3651913	.262
		.10	.2184000	.3651913	.566
	.10	.00	-1.2130000*	.3651913	.011
		.05	-.6593333	.3651913	.109
		.07	-.2184000	.3651913	.566

Table E-1.6a Day 7; BCR2 ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co2	Between Groups	.735	3	.245	6.884	.013
	Within Groups	.285	8	.036		
	Total	1.019	11			
Cr2	Between Groups	9.355	3	3.118	10.439	.004
	Within Groups	2.390	8	.299		
	Total	11.744	11			
Cu2	Between Groups	172.092	3	57.364	236.904	.000
	Within Groups	1.937	8	.242		
	Total	174.029	11			
Mn2	Between Groups	8126.045	3	2708.682	5.487	.024
	Within Groups	3949.555	8	493.694		
	Total	12075.600	11			
Ni2	Between Groups	2.168	3	.723	29.902	.000
	Within Groups	.193	8	.024		
	Total	2.361	11			
Pb2	Between Groups	26.000	3	8.667	10.007	.004
	Within Groups	6.929	8	.866		
	Total	32.928	11			
Zn2	Between Groups	15.703	3	5.234	12.730	.002
	Within Groups	3.289	8	.411		
	Total	18.993	11			

Table E-1.6b Day 7; BCR2 Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co2	.00	.05	-.0096333	.1539898	.952
		.07	-.3608000*	.1539898	.047
		.10	-.5858333*	.1539898	.005
	.05	.00	.0096333	.1539898	.952
		.07	-.3511667	.1539898	.052
		.10	-.5762000*	.1539898	.006
	.07	.00	.3608000*	.1539898	.047
		.05	.3511667	.1539898	.052
		.10	-.2250333	.1539898	.182
	.10	.00	.5858333*	.1539898	.005
		.05	.5762000*	.1539898	.006
		.07	.2250333	.1539898	.182
Cr2	.00	.05	1.2622000*	.4462551	.022
		.07	1.8481000*	.4462551	.003
		.10	2.3705333*	.4462551	.001
	.05	.00	-1.2622000*	.4462551	.022
		.07	.5859000	.4462551	.226
		.10	1.1083333*	.4462551	.038
	.07	.00	-1.8481000*	.4462551	.003
		.05	-.5859000	.4462551	.226
		.10	.5224333	.4462551	.275
	.10	.00	-2.3705333*	.4462551	.001
		.05	-1.1083333*	.4462551	.038
		.07	-.5224333	.4462551	.275
Cu2	.00	.05	6.9945333*	.4017804	.000
		.07	8.9120667*	.4017804	.000
		.10	9.5221000*	.4017804	.000
	.05	.00	-6.9945333*	.4017804	.000
		.07	1.9175333*	.4017804	.001
		.10	2.5275667*	.4017804	.000
	.07	.00	-8.9120667*	.4017804	.000
		.05	-1.9175333*	.4017804	.001
		.10	.6100333	.4017804	.167
	.10	.00	-9.5221000*	.4017804	.000
		.05	-2.5275667*	.4017804	.000
		.07	-.6100333	.4017804	.167
Mn2	.00	.05	59.4422000*	18.1419281	.011
		.07	64.9194333*	18.1419281	.007
		.10	53.8928333*	18.1419281	.018
	.05	.00	-59.4422000*	18.1419281	.011
		.07	5.4772333	18.1419281	.770
		.10	-5.5493667	18.1419281	.767
	.07	.00	-64.9194333*	18.1419281	.007
		.05	-5.4772333	18.1419281	.770
		.10	-11.0266000	18.1419281	.560
	.10	.00	-53.8928333*	18.1419281	.018
		.05	5.5493667	18.1419281	.767
		.07	11.0266000	18.1419281	.560

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni2	.00	.05	-.3887667*	.1269245	.016
		.07	-.8639000*	.1269245	.000
		.10	-1.0989000*	.1269245	.000
	.05	.00	.3887667*	.1269245	.016
		.07	-.4751333*	.1269245	.006
		.10	-.7101333*	.1269245	.001
	.07	.00	.8639000*	.1269245	.000
		.05	.4751333*	.1269245	.006
		.10	-.2350000	.1269245	.101
	.10	.00	1.0989000*	.1269245	.000
		.05	.7101333*	.1269245	.001
		.07	.2350000	.1269245	.101
Pb2	.00	.05	1.0844333	.7598579	.191
		.07	3.0004667*	.7598579	.004
		.10	3.6854000*	.7598579	.001
	.05	.00	-1.0844333	.7598579	.191
		.07	1.9160333*	.7598579	.036
		.10	2.6009667*	.7598579	.009
	.07	.00	-3.0004667*	.7598579	.004
		.05	-1.9160333*	.7598579	.036
		.10	.6849333	.7598579	.394
	.10	.00	-3.6854000*	.7598579	.001
		.05	-2.6009667*	.7598579	.009
		.07	-.6849333	.7598579	.394
Zn2	.00	.05	-1.0954667	.5235625	.070
		.07	-3.0380667*	.5235625	.000
		.10	-2.1942333*	.5235625	.003
	.05	.00	1.0954667	.5235625	.070
		.07	-1.9426000*	.5235625	.006
		.10	-1.0987667	.5235625	.069
	.07	.00	3.0380667*	.5235625	.000
		.05	1.9426000*	.5235625	.006
		.10	.8438333	.5235625	.146
	.10	.00	2.1942333*	.5235625	.003
		.05	1.0987667	.5235625	.069
		.07	-.8438333	.5235625	.146

Table E-1.7a Day 7; BCR3 ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co3	Between Groups	10.274	3	3.425	1.693	.245
	Within Groups	16.183	8	2.023		
	Total	26.457	11			
Cr3	Between Groups	31.325	3	10.442	5.215	.028
	Within Groups	16.018	8	2.002		
	Total	47.343	11			
Cu3	Between Groups	134.021	3	44.674	8.940	.006
	Within Groups	39.977	8	4.997		
	Total	173.998	11			
Mn3	Between Groups	594.041	3	198.014	14.621	.001
	Within Groups	108.343	8	13.543		
	Total	702.384	11			
Ni3	Between Groups	3.256	3	1.085	7.622	.010
	Within Groups	1.139	8	.142		
	Total	4.395	11			
Pb3	Between Groups	18.132	3	6.044	43.118	.000
	Within Groups	1.121	8	.140		
	Total	19.253	11			
Zn3	Between Groups	12.619	3	4.206	7.063	.012
	Within Groups	4.764	8	.596		
	Total	17.384	11			

Table E-1.7b Day 7; BCR3 Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co3	.00	.05	-.6688000	1.1612707	.581
		.07	-2.1337667	1.1612707	.103
		.10	-2.1132333	1.1612707	.106
	.05	.00	.6688000	1.1612707	.581
		.07	-1.4649667	1.1612707	.243
		.10	-1.4444333	1.1612707	.249
	.07	.00	2.1337667	1.1612707	.103
		.05	1.4649667	1.1612707	.243
		.10	.0205333	1.1612707	.986
	.10	.00	2.1132333	1.1612707	.106
		.05	1.4444333	1.1612707	.249
		.07	-.0205333	1.1612707	.986
Cr3	.00	.05	-1.7016000	1.1553530	.179
		.07	-3.4394333*	1.1553530	.018
		.10	-4.1703667*	1.1553530	.007
	.05	.00	1.7016000	1.1553530	.179
		.07	-1.7378333	1.1553530	.171
		.10	-2.4687667	1.1553530	.065
	.07	.00	3.4394333*	1.1553530	.018
		.05	1.7378333	1.1553530	.171
		.10	-.7309333	1.1553530	.545
	.10	.00	4.1703667*	1.1553530	.007
		.05	2.4687667	1.1553530	.065
		.07	.7309333	1.1553530	.545
Cu3	.00	.05	-5.1189000*	1.8252113	.023
		.07	-8.0158000*	1.8252113	.002
		.10	-8.3320333*	1.8252113	.002
	.05	.00	5.1189000*	1.8252113	.023
		.07	-2.8969000	1.8252113	.151
		.10	-3.2131333	1.8252113	.116
	.07	.00	8.0158000*	1.8252113	.002
		.05	2.8969000	1.8252113	.151
		.10	-.3162333	1.8252113	.867
	.10	.00	8.3320333*	1.8252113	.002
		.05	3.2131333	1.8252113	.116
		.07	.3162333	1.8252113	.867
Mn3	.00	.05	-9.1865667*	3.0047605	.016
		.07	-14.7878333*	3.0047605	.001
		.10	-18.7327333*	3.0047605	.000
	.05	.00	9.1865667*	3.0047605	.016
		.07	-5.6012667	3.0047605	.099
		.10	-9.5461667*	3.0047605	.013
	.07	.00	14.7878333*	3.0047605	.001
		.05	5.6012667	3.0047605	.099
		.10	-3.9449000	3.0047605	.226
	.10	.00	18.7327333*	3.0047605	.000
		.05	9.5461667*	3.0047605	.013
		.07	3.9449000	3.0047605	.226

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni3	.00	.05	-.6043333	.3080986	.085
		.07	-1.1059667*	.3080986	.007
		.10	-1.3634000*	.3080986	.002
	.05	.00	.6043333	.3080986	.085
		.07	-.5016333	.3080986	.142
		.10	-.7590667*	.3080986	.039
	.07	.00	1.1059667*	.3080986	.007
		.05	.5016333	.3080986	.142
		.10	-.2574333	.3080986	.428
	.10	.00	1.3634000*	.3080986	.002
		.05	.7590667*	.3080986	.039
		.07	.2574333	.3080986	.428
Pb3	.00	.05	-1.9806667*	.3056932	.000
		.07	-2.1229000*	.3056932	.000
		.10	-3.4422333*	.3056932	.000
	.05	.00	1.9806667*	.3056932	.000
		.07	-.1422333	.3056932	.654
		.10	-1.4615667*	.3056932	.001
	.07	.00	2.1229000*	.3056932	.000
		.05	.1422333	.3056932	.654
		.10	-1.3193333*	.3056932	.003
	.10	.00	3.4422333*	.3056932	.000
		.05	1.4615667*	.3056932	.001
		.07	1.3193333*	.3056932	.003
Zn3	.00	.05	-1.3316333	.6301055	.068
		.07	-2.0653333*	.6301055	.011
		.10	-2.7710333*	.6301055	.002
	.05	.00	1.3316333	.6301055	.068
		.07	-.7337000	.6301055	.278
		.10	-1.4394000	.6301055	.052
	.07	.00	2.0653333*	.6301055	.011
		.05	.7337000	.6301055	.278
		.10	-.7057000	.6301055	.295
	.10	.00	2.7710333*	.6301055	.002
		.05	1.4394000	.6301055	.052
		.07	.7057000	.6301055	.295

Table E-1.8a Day 7; RES ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co4	Between Groups	1.706	3	.569	3.583	.066
	Within Groups	1.270	8	.159		
	Total	2.975	11			
Cr4	Between Groups	112.755	3	37.585	1.057	.419
	Within Groups	284.474	8	35.559		
	Total	397.229	11			
Cu4	Between Groups	21.456	3	7.152	4.730	.035
	Within Groups	12.095	8	1.512		
	Total	33.551	11			
Mn4	Between Groups	30.057	3	10.019	.132	.938
	Within Groups	606.738	8	75.842		
	Total	636.795	11			
Ni4	Between Groups	.762	3	.254	.443	.729
	Within Groups	4.583	8	.573		
	Total	5.345	11			
Pb4	Between Groups	1.395	3	.465	1.561	.273
	Within Groups	2.383	8	.298		
	Total	3.778	11			
Zn4	Between Groups	113.233	3	37.744	2.470	.136
	Within Groups	122.250	8	15.281		
	Total	235.483	11			

Table E-1.8b Day 7; RES Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co4	.00	.05	.7608667*	.3252779	.047
		.07	.9286333*	.3252779	.021
		.10	.8872667*	.3252779	.026
	.05	.00	-.7608667*	.3252779	.047
		.07	.1677667	.3252779	.620
		.10	.1264000	.3252779	.708
	.07	.00	-.9286333*	.3252779	.021
		.05	-.1677667	.3252779	.620
		.10	-.0413667	.3252779	.902
	.10	.00	-.8872667*	.3252779	.026
		.05	-.1264000	.3252779	.708
		.07	.0413667	.3252779	.902
Cr4	.00	.05	-4.4143333	4.8688996	.391
		.07	-7.5467000	4.8688996	.160
		.10	-7.4191333	4.8688996	.166
	.05	.00	4.4143333	4.8688996	.391
		.07	-3.1323667	4.8688996	.538
		.10	-3.0048000	4.8688996	.554
	.07	.00	7.5467000	4.8688996	.160
		.05	3.1323667	4.8688996	.538
		.10	.1275667	4.8688996	.980
	.10	.00	7.4191333	4.8688996	.166
		.05	3.0048000	4.8688996	.554
		.07	-.1275667	4.8688996	.980
Cu4	.00	.05	3.0980000*	1.0039651	.015
		.07	2.8024667*	1.0039651	.024
		.10	3.2862333*	1.0039651	.011
	.05	.00	-3.0980000*	1.0039651	.015
		.07	-.2955333	1.0039651	.776
		.10	.1882333	1.0039651	.856
	.07	.00	-2.8024667*	1.0039651	.024
		.05	.2955333	1.0039651	.776
		.10	.4837667	1.0039651	.643
	.10	.00	-3.2862333*	1.0039651	.011
		.05	-.1882333	1.0039651	.856
		.07	-.4837667	1.0039651	.643
Mn4	.00	.05	2.4920667	7.1106635	.735
		.07	3.5238000	7.1106635	.634
		.10	4.1515667	7.1106635	.575
	.05	.00	-2.4920667	7.1106635	.735
		.07	1.0317333	7.1106635	.888
		.10	1.6595000	7.1106635	.821
	.07	.00	-3.5238000	7.1106635	.634
		.05	-1.0317333	7.1106635	.888
		.10	.6277667	7.1106635	.932
	.10	.00	-4.1515667	7.1106635	.575
		.05	-1.6595000	7.1106635	.821
		.07	-.6277667	7.1106635	.932

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni4	.00	.05	.5719333	.6180075	.382
		.07	.4989000	.6180075	.443
		.10	.0737667	.6180075	.908
	.05	.00	-.5719333	.6180075	.382
		.07	-.0730333	.6180075	.909
		.10	-.4981667	.6180075	.443
	.07	.00	-.4989000	.6180075	.443
		.05	.0730333	.6180075	.909
		.10	-.4251333	.6180075	.511
	.10	.00	-.0737667	.6180075	.908
		.05	.4981667	.6180075	.443
		.07	.4251333	.6180075	.511
Pb4	.00	.05	-.4563667	.4456191	.336
		.07	-.8390667	.4456191	.096
		.10	-.8182667	.4456191	.104
	.05	.00	.4563667	.4456191	.336
		.07	-.3827000	.4456191	.415
		.10	-.3619000	.4456191	.440
	.07	.00	.8390667	.4456191	.096
		.05	.3827000	.4456191	.415
		.10	.0208000	.4456191	.964
	.10	.00	.8182667	.4456191	.104
		.05	.3619000	.4456191	.440
		.07	-.0208000	.4456191	.964
Zn4	.00	.05	-1.3389333	3.1917852	.686
		.07	-3.7223667	3.1917852	.277
		.10	4.7040000	3.1917852	.179
	.05	.00	1.3389333	3.1917852	.686
		.07	-2.3834333	3.1917852	.477
		.10	6.0429333	3.1917852	.095
	.07	.00	3.7223667	3.1917852	.277
		.05	2.3834333	3.1917852	.477
		.10	8.4263667*	3.1917852	.030
	.10	.00	-4.7040000	3.1917852	.179
		.05	-6.0429333	3.1917852	.095
		.07	-8.4263667*	3.1917852	.030

Table E-1.9a Day 15; BCR1 ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co1	Between Groups	.405	3	.135	8.978	.006
	Within Groups	.120	8	.015		
	Total	.525	11			
Cr1	Between Groups	.169	3	.056	4.371	.042
	Within Groups	.103	8	.013		
	Total	.273	11			
Cu1	Between Groups	72.597	3	24.199	468.653	.000
	Within Groups	.413	8	.052		
	Total	73.010	11			
Mn1	Between Groups	7881.471	3	2627.157	7.332	.011
	Within Groups	2866.487	8	358.311		
	Total	10747.959	11			
Ni1	Between Groups	1.430	3	.477	16.752	.001
	Within Groups	.228	8	.028		
	Total	1.658	11			
Pb1	Between Groups	.668	3	.223	3.248	.081
	Within Groups	.548	8	.069		
	Total	1.217	11			
Zn1	Between Groups	6.182	3	2.061	11.618	.003
	Within Groups	1.419	8	.177		
	Total	7.601	11			

Table E-1.9b Day 15; BCR1 Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co1	.00	.05	-.0283333	.1001126	.784
		.07	.2456333*	.1001126	.040
		.10	.4180333*	.1001126	.003
	.05	.00	.0283333	.1001126	.784
		.07	.2739667*	.1001126	.026
		.10	.4463667*	.1001126	.002
	.07	.00	-.2456333*	.1001126	.040
		.05	-.2739667*	.1001126	.026
		.10	.1724000	.1001126	.123
	.10	.00	-.4180333*	.1001126	.003
		.05	-.4463667*	.1001126	.002
		.07	-.1724000	.1001126	.123
Cr1	.00	.05	-.2295000*	.0928141	.039
		.07	-.0121333	.0928141	.899
		.10	.0950667	.0928141	.336
	.05	.00	.2295000*	.0928141	.039
		.07	.2173667*	.0928141	.047
		.10	.3245667*	.0928141	.008
	.07	.00	.0121333	.0928141	.899
		.05	-.2173667*	.0928141	.047
		.10	.1072000	.0928141	.281
	.10	.00	-.0950667	.0928141	.336
		.05	-.3245667*	.0928141	.008
		.07	-.1072000	.0928141	.281
Cu1	.00	.05	5.5187333*	.1855362	.000
		.07	5.7116000*	.1855362	.000
		.10	5.7962333*	.1855362	.000
	.05	.00	-5.5187333*	.1855362	.000
		.07	.1928667	.1855362	.329
		.10	.2775000	.1855362	.173
	.07	.00	-5.7116000*	.1855362	.000
		.05	-.1928667	.1855362	.329
		.10	.0846333	.1855362	.660
	.10	.00	-5.7962333*	.1855362	.000
		.05	-.2775000	.1855362	.173
		.07	-.0846333	.1855362	.660
Mn1	.00	.05	-67.8721333*	15.4555473	.002
		.07	-51.6555667*	15.4555473	.010
		.10	-27.4854667	15.4555473	.113
	.05	.00	67.8721333*	15.4555473	.002
		.07	16.2165667	15.4555473	.325
		.10	40.3866667*	15.4555473	.031
	.07	.00	51.6555667*	15.4555473	.010
		.05	-16.2165667	15.4555473	.325
		.10	24.1701000	15.4555473	.156
	.10	.00	27.4854667	15.4555473	.113
		.05	-40.3866667*	15.4555473	.031
		.07	-24.1701000	15.4555473	.156

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni1	.00	.05	-.4710333*	.1377429	.009
		.07	-.9468667*	.1377429	.000
		.10	-.6675000*	.1377429	.001
	.05	.00	.4710333*	.1377429	.009
		.07	-.4758333*	.1377429	.009
		.10	-.1964667	.1377429	.192
	.07	.00	.9468667*	.1377429	.000
		.05	.4758333*	.1377429	.009
		.10	.2793667	.1377429	.077
	.10	.00	.6675000*	.1377429	.001
		.05	.1964667	.1377429	.192
		.07	-.2793667	.1377429	.077
Pb1	.00	.05	.2212000	.2137899	.331
		.07	.6158667*	.2137899	.020
		.10	.0857000	.2137899	.699
	.05	.00	-.2212000	.2137899	.331
		.07	.3946667	.2137899	.102
		.10	-.1355000	.2137899	.544
	.07	.00	-.6158667*	.2137899	.020
		.05	-.3946667	.2137899	.102
		.10	-.5301667*	.2137899	.038
	.10	.00	-.0857000	.2137899	.699
		.05	.1355000	.2137899	.544
		.07	.5301667*	.2137899	.038
Zn1	.00	.05	.8516000*	.3438675	.038
		.07	1.3017333*	.3438675	.005
		.10	1.9755667*	.3438675	.000
	.05	.00	-.8516000*	.3438675	.038
		.07	.4501333	.3438675	.227
		.10	1.1239667*	.3438675	.011
	.07	.00	-1.3017333*	.3438675	.005
		.05	-.4501333	.3438675	.227
		.10	.6738333	.3438675	.086
	.10	.00	-1.9755667*	.3438675	.000
		.05	-1.1239667*	.3438675	.011
		.07	-.6738333	.3438675	.086

Table E-1.10a Day 15; BCR2 ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co2	Between Groups	5.999	3	2.000	16.097	.001
	Within Groups	.994	8	.124		
	Total	6.992	11			
Cr2	Between Groups	21.201	3	7.067	16.624	.001
	Within Groups	3.401	8	.425		
	Total	24.601	11			
Cu2	Between Groups	131.101	3	43.700	479.912	.000
	Within Groups	.728	8	.091		
	Total	131.829	11			
Mn2	Between Groups	17002.545	3	5667.515	6.603	.015
	Within Groups	6866.470	8	858.309		
	Total	23869.016	11			
Ni2	Between Groups	3.184	3	1.061	13.839	.002
	Within Groups	.613	8	.077		
	Total	3.797	11			
Pb2	Between Groups	24.855	3	8.285	10.955	.003
	Within Groups	6.050	8	.756		
	Total	30.905	11			
Zn2	Between Groups	22.041	3	7.347	22.522	.000
	Within Groups	2.610	8	.326		
	Total	24.651	11			

Table E-1.10b Day 15; BCR2 Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co2	.00	.05	-.9778000*	.2877672	.009
		.07	-1.9867333*	.2877672	.000
		.10	-1.1737000*	.2877672	.004
	.05	.00	.9778000*	.2877672	.009
		.07	-1.0089333*	.2877672	.008
		.10	-.1959000	.2877672	.515
	.07	.00	1.9867333*	.2877672	.000
		.05	1.0089333*	.2877672	.008
		.10	.8130333*	.2877672	.022
	.10	.00	1.1737000*	.2877672	.004
		.05	.1959000	.2877672	.515
		.07	-.8130333*	.2877672	.022
Cr2	.00	.05	2.1831667*	.5323467	.003
		.07	2.8368333*	.5323467	.001
		.10	3.5541667*	.5323467	.000
	.05	.00	-2.1831667*	.5323467	.003
		.07	.6536667	.5323467	.254
		.10	1.3710000*	.5323467	.033
	.07	.00	-2.8368333*	.5323467	.001
		.05	-.6536667	.5323467	.254
		.10	.7173333	.5323467	.215
	.10	.00	-3.5541667*	.5323467	.000
		.05	-1.3710000*	.5323467	.033
		.07	-.7173333	.5323467	.215
Cu2	.00	.05	6.4420000*	.2463857	.000
		.07	7.4737000*	.2463857	.000
		.10	8.4497000*	.2463857	.000
	.05	.00	-6.4420000*	.2463857	.000
		.07	1.0317000*	.2463857	.003
		.10	2.0077000*	.2463857	.000
	.07	.00	-7.4737000*	.2463857	.000
		.05	-1.0317000*	.2463857	.003
		.10	.9760000*	.2463857	.004
	.10	.00	-8.4497000*	.2463857	.000
		.05	-2.0077000*	.2463857	.000
		.07	-.9760000*	.2463857	.004
Mn2	.00	.05	21.7170000	23.9208250	.390
		.07	-40.6342667	23.9208250	.128
		.10	63.5897000*	23.9208250	.029
	.05	.00	-21.7170000	23.9208250	.390
		.07	-62.3512667*	23.9208250	.031
		.10	41.8727000	23.9208250	.118
	.07	.00	40.6342667	23.9208250	.128
		.05	62.3512667*	23.9208250	.031
		.10	104.2239667*	23.9208250	.002
	.10	.00	-63.5897000*	23.9208250	.029
		.05	-41.8727000	23.9208250	.118
		.07	-1.0422397E2	23.9208250	.002

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni2	.00	.05	-.7381667*	.2261054	.011
		.07	-1.3527333*	.2261054	.000
		.10	-1.1347667*	.2261054	.001
	.05	.00	.7381667*	.2261054	.011
		.07	-.6145667*	.2261054	.026
		.10	-.3966000	.2261054	.118
	.07	.00	1.3527333*	.2261054	.000
		.05	.6145667*	.2261054	.026
		.10	.2179667	.2261054	.363
	.10	.00	1.1347667*	.2261054	.001
		.05	.3966000	.2261054	.118
		.07	-.2179667	.2261054	.363
Pb2	.00	.05	1.3704667	.7100514	.090
		.07	-1.2211333	.7100514	.124
		.10	2.6118333*	.7100514	.006
	.05	.00	-1.3704667	.7100514	.090
		.07	-2.5916000*	.7100514	.006
		.10	1.2413667	.7100514	.119
	.07	.00	1.2211333	.7100514	.124
		.05	2.5916000*	.7100514	.006
		.10	3.8329667*	.7100514	.001
	.10	.00	-2.6118333*	.7100514	.006
		.05	-1.2413667	.7100514	.119
		.07	-3.8329667*	.7100514	.001
Zn2	.00	.05	-1.7150000*	.4663469	.006
		.07	-3.8097333*	.4663469	.000
		.10	-2.1380000*	.4663469	.002
	.05	.00	1.7150000*	.4663469	.006
		.07	-2.0947333*	.4663469	.002
		.10	-.4230000	.4663469	.391
	.07	.00	3.8097333*	.4663469	.000
		.05	2.0947333*	.4663469	.002
		.10	1.6717333*	.4663469	.007
	.10	.00	2.1380000*	.4663469	.002
		.05	.4230000	.4663469	.391
		.07	-1.6717333*	.4663469	.007

Table E-1.11a Day 15; BCR3 ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co3	Between Groups	36.927	3	12.309	2.973	.097
	Within Groups	33.118	8	4.140		
	Total	70.045	11			
Cr3	Between Groups	45.994	3	15.331	3.898	.055
	Within Groups	31.468	8	3.933		
	Total	77.462	11			
Cu3	Between Groups	108.280	3	36.093	6.489	.016
	Within Groups	44.497	8	5.562		
	Total	152.777	11			
Mn3	Between Groups	1909.705	3	636.568	3.068	.091
	Within Groups	1659.990	8	207.499		
	Total	3569.695	11			
Ni3	Between Groups	.114	3	.038	6.401	.016
	Within Groups	.047	8	.006		
	Total	.161	11			
Pb3	Between Groups	18.132	3	6.044	43.118	.000
	Within Groups	1.121	8	.140		
	Total	19.253	11			
Zn3	Between Groups	23.043	3	7.681	2.346	.149
	Within Groups	26.194	8	3.274		
	Total	49.236	11			

Table E-1.11b Day 15; BCR3 Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co3	.00	.05	-1.9150667	1.6612679	.282
		.07	-3.5393667	1.6612679	.066
		.10	-4.6539333*	1.6612679	.023
	.05	.00	1.9150667	1.6612679	.282
		.07	-1.6243000	1.6612679	.357
		.10	-2.7388667	1.6612679	.138
	.07	.00	3.5393667	1.6612679	.066
		.05	1.6243000	1.6612679	.357
		.10	-1.1145667	1.6612679	.521
	.10	.00	4.6539333*	1.6612679	.023
		.05	2.7388667	1.6612679	.138
		.07	1.1145667	1.6612679	.521
Cr3	.00	.05	-.9348000	1.6193612	.580
		.07	-2.0944000	1.6193612	.232
		.10	-5.1938000*	1.6193612	.012
	.05	.00	.9348000	1.6193612	.580
		.07	-1.1596000	1.6193612	.494
		.10	-4.2590000*	1.6193612	.030
	.07	.00	2.0944000	1.6193612	.232
		.05	1.1596000	1.6193612	.494
		.10	-3.0994000	1.6193612	.092
	.10	.00	5.1938000*	1.6193612	.012
		.05	4.2590000*	1.6193612	.030
		.07	3.0994000	1.6193612	.092
Cu3	.00	.05	-4.6040667*	1.9256285	.044
		.07	-7.1022667*	1.9256285	.006
		.10	-7.5778333*	1.9256285	.004
	.05	.00	4.6040667*	1.9256285	.044
		.07	-2.4982000	1.9256285	.231
		.10	-2.9737667	1.9256285	.161
	.07	.00	7.1022667*	1.9256285	.006
		.05	2.4982000	1.9256285	.231
		.10	-.4755667	1.9256285	.811
	.10	.00	7.5778333*	1.9256285	.004
		.05	2.9737667	1.9256285	.161
		.07	.4755667	1.9256285	.811
Mn3	.00	.05	-24.3923667	11.7614848	.072
		.07	-25.8011667	11.7614848	.060
		.10	-33.6890667*	11.7614848	.021
	.05	.00	24.3923667	11.7614848	.072
		.07	-1.4088000	11.7614848	.908
		.10	-9.2967000	11.7614848	.452
	.07	.00	25.8011667	11.7614848	.060
		.05	1.4088000	11.7614848	.908
		.10	-7.8879000	11.7614848	.521
	.10	.00	33.6890667*	11.7614848	.021
		.05	9.2967000	11.7614848	.452
		.07	7.8879000	11.7614848	.521

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni3	.00	.05	-.1793667*	.0627759	.021
		.07	.0321333	.0627759	.623
		.10	-.1748333*	.0627759	.024
	.05	.00	.1793667*	.0627759	.021
		.07	.2115000*	.0627759	.010
		.10	.0045333	.0627759	.944
	.07	.00	-.0321333	.0627759	.623
		.05	-.2115000*	.0627759	.010
		.10	-.2069667*	.0627759	.011
	.10	.00	.1748333*	.0627759	.024
		.05	-.0045333	.0627759	.944
		.07	.2069667*	.0627759	.011
Pb3	.00	.05	-1.9806667*	.3056932	.000
		.07	-2.1229000*	.3056932	.000
		.10	-3.4422333*	.3056932	.000
	.05	.00	1.9806667*	.3056932	.000
		.07	-.1422333	.3056932	.654
		.10	-1.4615667*	.3056932	.001
	.07	.00	2.1229000*	.3056932	.000
		.05	.1422333	.3056932	.654
		.10	-1.3193333*	.3056932	.003
	.10	.00	3.4422333*	.3056932	.000
		.05	1.4615667*	.3056932	.001
		.07	1.3193333*	.3056932	.003
Zn3	.00	.05	-1.6667667	1.4774330	.292
		.07	-2.0156000	1.4774330	.210
		.10	-3.9008000*	1.4774330	.030
	.05	.00	1.6667667	1.4774330	.292
		.07	-.3488333	1.4774330	.819
		.10	-2.2340333	1.4774330	.169
	.07	.00	2.0156000	1.4774330	.210
		.05	.3488333	1.4774330	.819
		.10	-1.8852000	1.4774330	.238
	.10	.00	3.9008000*	1.4774330	.030
		.05	2.2340333	1.4774330	.169
		.07	1.8852000	1.4774330	.238

Table E-1.12a Day 15; RES ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co4	Between Groups	5.715	3	1.905	17.201	.001
	Within Groups	.886	8	.111		
	Total	6.601	11			
Cr4	Between Groups	105.437	3	35.146	4.097	.049
	Within Groups	68.624	8	8.578		
	Total	174.062	11			
Cu4	Between Groups	73.027	3	24.342	14.031	.001
	Within Groups	13.879	8	1.735		
	Total	86.906	11			
Mn4	Between Groups	10033.893	3	3344.631	30.854	.000
	Within Groups	867.208	8	108.401		
	Total	10901.101	11			
Ni4	Between Groups	44.686	3	14.895	16.136	.001
	Within Groups	7.385	8	.923		
	Total	52.070	11			
Pb4	Between Groups	1116.640	3	372.213	1.280	.345
	Within Groups	2327.147	8	290.893		
	Total	3443.787	11			
Zn4	Between Groups	437.536	3	145.845	21.653	.000
	Within Groups	53.884	8	6.735		
	Total	491.420	11			

Table E-1.12b Day 15; RES Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co4	.00	.05	.9144333*	.2717223	.010
		.07	1.9474333*	.2717223	.000
		.10	1.0468000*	.2717223	.005
	.05	.00	-.9144333*	.2717223	.010
		.07	1.0330000*	.2717223	.005
		.10	.1323667	.2717223	.639
	.07	.00	-1.9474333*	.2717223	.000
		.05	-1.0330000*	.2717223	.005
		.10	-.9006333*	.2717223	.011
	.10	.00	-1.0468000*	.2717223	.005
		.05	-.1323667	.2717223	.639
		.07	.9006333*	.2717223	.011
Cr4	.00	.05	-3.6577667	2.3913765	.165
		.07	4.7043333	2.3913765	.085
		.10	.3138333	2.3913765	.899
	.05	.00	3.6577667	2.3913765	.165
		.07	8.3621000*	2.3913765	.008
		.10	3.9716000	2.3913765	.135
	.07	.00	-4.7043333	2.3913765	.085
		.05	-8.3621000*	2.3913765	.008
		.10	-4.3905000	2.3913765	.104
	.10	.00	-.3138333	2.3913765	.899
		.05	-3.9716000	2.3913765	.135
		.07	4.3905000	2.3913765	.104
Cu4	.00	.05	2.8087000*	1.0754414	.031
		.07	6.8841333*	1.0754414	.000
		.10	3.9420333*	1.0754414	.006
	.05	.00	-2.8087000*	1.0754414	.031
		.07	4.0754333*	1.0754414	.005
		.10	1.1333333	1.0754414	.323
	.07	.00	-6.8841333*	1.0754414	.000
		.05	-4.0754333*	1.0754414	.005
		.10	-2.9421000*	1.0754414	.026
	.10	.00	-3.9420333*	1.0754414	.006
		.05	-1.1333333	1.0754414	.323
		.07	2.9421000*	1.0754414	.026
Mn4	.00	.05	30.2891667*	8.5010199	.007
		.07	80.6068333*	8.5010199	.000
		.10	43.1882333*	8.5010199	.001
	.05	.00	-30.2891667*	8.5010199	.007
		.07	50.3176667*	8.5010199	.000
		.10	12.8990667	8.5010199	.168
	.07	.00	-80.6068333*	8.5010199	.000
		.05	-50.3176667*	8.5010199	.000
		.10	-37.4186000*	8.5010199	.002
	.10	.00	-43.1882333*	8.5010199	.001
		.05	-12.8990667	8.5010199	.168
		.07	37.4186000*	8.5010199	.002

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni4	.00	.05	-4.4694000*	.7844669	.000
		.07	.2663000	.7844669	.743
		.10	-.4094667	.7844669	.616
	.05	.00	4.4694000*	.7844669	.000
		.07	4.7357000*	.7844669	.000
		.10	4.0599333*	.7844669	.001
	.07	.00	-.2663000	.7844669	.743
		.05	-4.7357000*	.7844669	.000
		.10	-.6757667	.7844669	.414
	.10	.00	.4094667	.7844669	.616
		.05	-4.0599333*	.7844669	.001
		.07	.6757667	.7844669	.414
Pb4	.00	.05	-20.9417667	13.9258362	.171
		.07	-10.4056333	13.9258362	.476
		.10	-24.7288000	13.9258362	.114
	.05	.00	20.9417667	13.9258362	.171
		.07	10.5361333	13.9258362	.471
		.10	-3.7870333	13.9258362	.793
	.07	.00	10.4056333	13.9258362	.476
		.05	-10.5361333	13.9258362	.471
		.10	-14.3231667	13.9258362	.334
	.10	.00	24.7288000	13.9258362	.114
		.05	3.7870333	13.9258362	.793
		.07	14.3231667	13.9258362	.334
Zn4	.00	.05	.7835333	2.1190344	.721
		.07	15.1213667*	2.1190344	.000
		.10	6.3956000*	2.1190344	.017
	.05	.00	-.7835333	2.1190344	.721
		.07	14.3378333*	2.1190344	.000
		.10	5.6120667*	2.1190344	.029
	.07	.00	-15.1213667*	2.1190344	.000
		.05	-14.3378333*	2.1190344	.000
		.10	-8.7257667*	2.1190344	.003
	.10	.00	-6.3956000*	2.1190344	.017
		.05	-5.6120667*	2.1190344	.029
		.07	8.7257667*	2.1190344	.003

Table E-1.13a Day 30; BCR1 ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co1	Between Groups	.630	3	.210	20.296	.000
	Within Groups	.083	8	.010		
	Total	.713	11			
Cr1	Between Groups	.057	3	.019	1.481	.292
	Within Groups	.103	8	.013		
	Total	.161	11			
Cu1	Between Groups	47.444	3	15.815	1388.857	.000
	Within Groups	.091	8	.011		
	Total	47.535	11			
Mn1	Between Groups	17323.911	3	5774.637	28.575	.000
	Within Groups	1616.668	8	202.084		
	Total	18940.579	11			
Ni1	Between Groups	1.603	3	.534	18.434	.001
	Within Groups	.232	8	.029		
	Total	1.835	11			
Pb1	Between Groups	1.715	3	.572	42.019	.000
	Within Groups	.109	8	.014		
	Total	1.824	11			
Zn1	Between Groups	6.496	3	2.165	8.255	.008
	Within Groups	2.099	8	.262		
	Total	8.595	11			

Table E-1.13b Day 30; BCR1 Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co1	.00	.05	-.1553667	.0830483	.098
		.07	.2066000*	.0830483	.038
		.10	.4553000*	.0830483	.001
	.05	.00	.1553667	.0830483	.098
		.07	.3619667*	.0830483	.002
		.10	.6106667*	.0830483	.000
	.07	.00	-.2066000*	.0830483	.038
		.05	-.3619667*	.0830483	.002
		.10	.2487000*	.0830483	.017
	.10	.00	-.4553000*	.0830483	.001
		.05	-.6106667*	.0830483	.000
		.07	-.2487000*	.0830483	.017
Cr1	.00	.05	.1329000	.0928594	.190
		.07	.0984333	.0928594	.320
		.10	.1904667	.0928594	.074
	.05	.00	-.1329000	.0928594	.190
		.07	-.0344667	.0928594	.720
		.10	.0575667	.0928594	.553
	.07	.00	-.0984333	.0928594	.320
		.05	.0344667	.0928594	.720
		.10	.0920333	.0928594	.351
	.10	.00	-.1904667	.0928594	.074
		.05	-.0575667	.0928594	.553
		.07	-.0920333	.0928594	.351
Cu1	.00	.05	4.5161000*	.0871278	.000
		.07	4.6160000*	.0871278	.000
		.10	4.6401000*	.0871278	.000
	.05	.00	-4.5161000*	.0871278	.000
		.07	.0999000	.0871278	.285
		.10	.1240000	.0871278	.192
	.07	.00	-4.6160000*	.0871278	.000
		.05	-.0999000	.0871278	.285
		.10	.0241000	.0871278	.789
	.10	.00	-4.6401000*	.0871278	.000
		.05	-.1240000	.0871278	.192
		.07	-.0241000	.0871278	.789
Mn1	.00	.05	-1.0626470E2	11.6069964	.000
		.07	-65.9609667*	11.6069964	.000
		.10	-62.4189667*	11.6069964	.001
	.05	.00	106.2647000*	11.6069964	.000
		.07	40.3037333*	11.6069964	.008
		.10	43.8457333*	11.6069964	.005
	.07	.00	65.9609667*	11.6069964	.000
		.05	-40.3037333*	11.6069964	.008
		.10	3.5420000	11.6069964	.768
	.10	.00	62.4189667*	11.6069964	.001
		.05	-43.8457333*	11.6069964	.005
		.07	-3.5420000	11.6069964	.768

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni1	.00	.05	-.7370000*	.1389996	.001
		.07	-.8749667*	.1389996	.000
		.10	-.8869000*	.1389996	.000
	.05	.00	.7370000*	.1389996	.001
		.07	-.1379667	.1389996	.350
		.10	-.1499000	.1389996	.312
	.07	.00	.8749667*	.1389996	.000
		.05	.1379667	.1389996	.350
		.10	-.0119333	.1389996	.934
	.10	.00	.8869000*	.1389996	.000
		.05	.1499000	.1389996	.312
		.07	.0119333	.1389996	.934
Pb1	.00	.05	.7037667*	.0952344	.000
		.07	.8333667*	.0952344	.000
		.10	.9877000*	.0952344	.000
	.05	.00	-.7037667*	.0952344	.000
		.07	.1296000	.0952344	.211
		.10	.2839333*	.0952344	.018
	.07	.00	-.8333667*	.0952344	.000
		.05	-.1296000	.0952344	.211
		.10	.1543333	.0952344	.144
	.10	.00	-.9877000*	.0952344	.000
		.05	-.2839333*	.0952344	.018
		.07	-.1543333	.0952344	.144
Zn1	.00	.05	.9472333	.4181842	.053
		.07	1.0688333*	.4181842	.034
		.10	2.0770333*	.4181842	.001
	.05	.00	-.9472333	.4181842	.053
		.07	.1216000	.4181842	.779
		.10	1.1298000*	.4181842	.027
	.07	.00	-1.0688333*	.4181842	.034
		.05	-.1216000	.4181842	.779
		.10	1.0082000*	.4181842	.042
	.10	.00	-2.0770333*	.4181842	.001
		.05	-1.1298000*	.4181842	.027
		.07	-1.0082000*	.4181842	.042

Table E-1.14a Day 30; BCR2 ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co2	Between Groups	6.789	3	2.263	13.746	.002
	Within Groups	1.317	8	.165		
	Total	8.106	11			
Cr2	Between Groups	24.753	3	8.251	8.159	.008
	Within Groups	8.090	8	1.011		
	Total	32.843	11			
Cu2	Between Groups	158.224	3	52.741	659.826	.000
	Within Groups	.639	8	.080		
	Total	158.864	11			
Mn2	Between Groups	10769.457	3	3589.819	2.251	.160
	Within Groups	12755.570	8	1594.446		
	Total	23525.027	11			
Ni2	Between Groups	4.024	3	1.341	8.635	.007
	Within Groups	1.243	8	.155		
	Total	5.266	11			
Pb2	Between Groups	101.107	3	33.702	3.901	.055
	Within Groups	69.109	8	8.639		
	Total	170.217	11			
Zn2	Between Groups	29.309	3	9.770	7.513	.010
	Within Groups	10.403	8	1.300		
	Total	39.712	11			

Table E-1.14b Day 30; BCR2 Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co2	.00	.05	-.8773000*	.3312996	.029
		.07	-2.0139667*	.3312996	.000
		.10	-.4174667	.3312996	.243
	.05	.00	.8773000*	.3312996	.029
		.07	-1.1366667*	.3312996	.009
		.10	.4598333	.3312996	.203
	.07	.00	2.0139667*	.3312996	.000
		.05	1.1366667*	.3312996	.009
		.10	1.5965000*	.3312996	.001
	.10	.00	.4174667	.3312996	.243
		.05	-.4598333	.3312996	.203
		.07	-1.5965000*	.3312996	.001
Cr2	.00	.05	1.0682000	.8210724	.229
		.07	3.1086333*	.8210724	.005
		.10	3.4776667*	.8210724	.003
	.05	.00	-1.0682000	.8210724	.229
		.07	2.0404333*	.8210724	.038
		.10	2.4094667*	.8210724	.019
	.07	.00	-3.1086333*	.8210724	.005
		.05	-2.0404333*	.8210724	.038
		.10	.3690333	.8210724	.665
	.10	.00	-3.4776667*	.8210724	.003
		.05	-2.4094667*	.8210724	.019
		.07	-.3690333	.8210724	.665
Cu2	.00	.05	6.4329667*	.2308426	.000
		.07	8.1481000*	.2308426	.000
		.10	9.4554333*	.2308426	.000
	.05	.00	-6.4329667*	.2308426	.000
		.07	1.7151333*	.2308426	.000
		.10	3.0224667*	.2308426	.000
	.07	.00	-8.1481000*	.2308426	.000
		.05	-1.7151333*	.2308426	.000
		.10	1.3073333*	.2308426	.000
	.10	.00	-9.4554333*	.2308426	.000
		.05	-3.0224667*	.2308426	.000
		.07	-1.3073333*	.2308426	.000
Mn2	.00	.05	42.5274667	32.6031314	.228
		.07	40.2354000	32.6031314	.252
		.10	84.6907667*	32.6031314	.032
	.05	.00	-42.5274667	32.6031314	.228
		.07	-2.2920667	32.6031314	.946
		.10	42.1633000	32.6031314	.232
	.07	.00	-40.2354000	32.6031314	.252
		.05	2.2920667	32.6031314	.946
		.10	44.4553667	32.6031314	.210
	.10	.00	-84.6907667*	32.6031314	.032
		.05	-42.1633000	32.6031314	.232
		.07	-44.4553667	32.6031314	.210

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni2	.00	.05	-1.2257333*	.3217960	.005
		.07	-1.3406667*	.3217960	.003
		.10	-1.4174667*	.3217960	.002
	.05	.00	1.2257333*	.3217960	.005
		.07	-.1149333	.3217960	.730
		.10	-.1917333	.3217960	.568
	.07	.00	1.3406667*	.3217960	.003
		.05	.1149333	.3217960	.730
		.10	-.0768000	.3217960	.817
	.10	.00	1.4174667*	.3217960	.002
		.05	.1917333	.3217960	.568
		.07	.0768000	.3217960	.817
Pb2	.00	.05	-2.1671000	2.3998134	.393
		.07	4.0959000	2.3998134	.126
		.10	4.8808333	2.3998134	.076
	.05	.00	2.1671000	2.3998134	.393
		.07	6.2630000*	2.3998134	.031
		.10	7.0479333*	2.3998134	.019
	.07	.00	-4.0959000	2.3998134	.126
		.05	-6.2630000*	2.3998134	.031
		.10	.7849333	2.3998134	.752
	.10	.00	-4.8808333	2.3998134	.076
		.05	-7.0479333*	2.3998134	.019
		.07	-.7849333	2.3998134	.752
Zn2	.00	.05	-4.1490333*	.9310702	.002
		.07	-3.3940667*	.9310702	.007
		.10	-2.5626333*	.9310702	.025
	.05	.00	4.1490333*	.9310702	.002
		.07	.7549667	.9310702	.441
		.10	1.5864000	.9310702	.127
	.07	.00	3.3940667*	.9310702	.007
		.05	-.7549667	.9310702	.441
		.10	.8314333	.9310702	.398
	.10	.00	2.5626333*	.9310702	.025
		.05	-1.5864000	.9310702	.127
		.07	-.8314333	.9310702	.398

Table E-1.15a Day 30; BCR3 ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co3	Between Groups	4.852	3	1.617	.589	.639
	Within Groups	21.982	8	2.748		
	Total	26.835	11			
Cr3	Between Groups	20.287	3	6.762	7.780	.009
	Within Groups	6.954	8	.869		
	Total	27.241	11			
Cu3	Between Groups	137.266	3	45.755	8.108	.008
	Within Groups	45.146	8	5.643		
	Total	182.413	11			
Mn3	Between Groups	169.480	3	56.493	10.084	.004
	Within Groups	44.820	8	5.602		
	Total	214.300	11			
Ni3	Between Groups	.060	3	.020	1.704	.243
	Within Groups	.094	8	.012		
	Total	.153	11			
Pb3	Between Groups	6.302	3	2.101	12.569	.002
	Within Groups	1.337	8	.167		
	Total	7.639	11			
Zn3	Between Groups	8.830	3	2.943	7.469	.010
	Within Groups	3.152	8	.394		
	Total	11.982	11			

Table E-1.15b Day 30; BCR3 Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co3	.00	.05	-1.3362333	1.3534639	.352
		.07	-1.7104667	1.3534639	.242
		.10	-1.0381333	1.3534639	.465
	.05	.00	1.3362333	1.3534639	.352
		.07	-.3742333	1.3534639	.789
		.10	.2981000	1.3534639	.831
	.07	.00	1.7104667	1.3534639	.242
		.05	.3742333	1.3534639	.789
		.10	.6723333	1.3534639	.633
	.10	.00	1.0381333	1.3534639	.465
		.05	-.2981000	1.3534639	.831
		.07	-.6723333	1.3534639	.633
Cr3	.00	.05	-1.2117000	.7612233	.150
		.07	-2.9889333*	.7612233	.004
		.10	-3.1293000*	.7612233	.003
	.05	.00	1.2117000	.7612233	.150
		.07	-1.7772333*	.7612233	.048
		.10	-1.9176000*	.7612233	.036
	.07	.00	2.9889333*	.7612233	.004
		.05	1.7772333*	.7612233	.048
		.10	-.1403667	.7612233	.858
	.10	.00	3.1293000*	.7612233	.003
		.05	1.9176000*	.7612233	.036
		.07	.1403667	.7612233	.858
Cu3	.00	.05	-7.0812333*	1.9396339	.006
		.07	-8.0570333*	1.9396339	.003
		.10	-8.1196000*	1.9396339	.003
	.05	.00	7.0812333*	1.9396339	.006
		.07	-.9758000	1.9396339	.628
		.10	-1.0383667	1.9396339	.607
	.07	.00	8.0570333*	1.9396339	.003
		.05	.9758000	1.9396339	.628
		.10	-.0625667	1.9396339	.975
	.10	.00	8.1196000*	1.9396339	.003
		.05	1.0383667	1.9396339	.607
		.07	.0625667	1.9396339	.975
Mn3	.00	.05	-5.2038000*	1.9326110	.027
		.07	-7.9792000*	1.9326110	.003
		.10	-10.0129667*	1.9326110	.001
	.05	.00	5.2038000*	1.9326110	.027
		.07	-2.7754000	1.9326110	.189
		.10	-4.8091667*	1.9326110	.038
	.07	.00	7.9792000*	1.9326110	.003
		.05	2.7754000	1.9326110	.189
		.10	-2.0337667	1.9326110	.323
	.10	.00	10.0129667*	1.9326110	.001
		.05	4.8091667*	1.9326110	.038
		.07	2.0337667	1.9326110	.323

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni3	.00	.05	-.0686000	.0882950	.460
		.07	-.1938667	.0882950	.059
		.10	-.1157667	.0882950	.226
	.05	.00	.0686000	.0882950	.460
		.07	-.1252667	.0882950	.194
		.10	-.0471667	.0882950	.608
	.07	.00	.1938667	.0882950	.059
		.05	.1252667	.0882950	.194
		.10	.0781000	.0882950	.402
	.10	.00	.1157667	.0882950	.226
		.05	.0471667	.0882950	.608
		.07	-.0781000	.0882950	.402
Pb3	.00	.05	-.6645667	.3338036	.082
		.07	-1.7875667*	.3338036	.001
		.10	-1.6073667*	.3338036	.001
	.05	.00	.6645667	.3338036	.082
		.07	-1.1230000*	.3338036	.010
		.10	-.9428000*	.3338036	.022
	.07	.00	1.7875667*	.3338036	.001
		.05	1.1230000*	.3338036	.010
		.10	.1802000	.3338036	.604
	.10	.00	1.6073667*	.3338036	.001
		.05	.9428000*	.3338036	.022
		.07	-.1802000	.3338036	.604
Zn3	.00	.05	-.9659667	.5125435	.096
		.07	-1.2372333*	.5125435	.042
		.10	-2.4067000*	.5125435	.002
	.05	.00	.9659667	.5125435	.096
		.07	-.2712667	.5125435	.611
		.10	-1.4407333*	.5125435	.023
	.07	.00	1.2372333*	.5125435	.042
		.05	.2712667	.5125435	.611
		.10	-1.1694667	.5125435	.052
	.10	.00	2.4067000*	.5125435	.002
		.05	1.4407333*	.5125435	.023
		.07	1.1694667	.5125435	.052

Table E-1.16a Day 30; RES ANOVA

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Co4	Between Groups	.659	3	.220	.501	.692
	Within Groups	3.504	8	.438		
	Total	4.163	11			
Cr4	Between Groups	76.688	3	25.563	.418	.745
	Within Groups	488.873	8	61.109		
	Total	565.561	11			
Cu4	Between Groups	35.828	3	11.943	3.334	.077
	Within Groups	28.654	8	3.582		
	Total	64.482	11			
Mn4	Between Groups	1120.690	3	373.563	.624	.619
	Within Groups	4787.936	8	598.492		
	Total	5908.626	11			
Ni4	Between Groups	3.449	3	1.150	1.201	.370
	Within Groups	7.656	8	.957		
	Total	11.105	11			
Pb4	Between Groups	2.311	3	.770	1.017	.434
	Within Groups	6.059	8	.757		
	Total	8.370	11			
Zn4	Between Groups	83.684	3	27.895	.868	.496
	Within Groups	257.093	8	32.137		
	Total	340.777	11			

Table E-1.16b Day 30; RES Post Hoc Tests

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Co4	.00	.05	.2347667	.5403902	.675
		.07	.5470333	.5403902	.341
		.10	-.0427000	.5403902	.939
	.05	.00	-.2347667	.5403902	.675
		.07	.3122667	.5403902	.579
		.10	-.2774667	.5403902	.622
	.07	.00	-.5470333	.5403902	.341
		.05	-.3122667	.5403902	.579
		.10	-.5897333	.5403902	.307
	.10	.00	.0427000	.5403902	.939
		.05	.2774667	.5403902	.622
		.07	.5897333	.5403902	.307
Cr4	.00	.05	6.0906333	6.3827421	.368
		.07	2.8654000	6.3827421	.665
		.10	6.0389000	6.3827421	.372
	.05	.00	-6.0906333	6.3827421	.368
		.07	-3.2252333	6.3827421	.627
		.10	-.0517333	6.3827421	.994
	.07	.00	-2.8654000	6.3827421	.665
		.05	3.2252333	6.3827421	.627
		.10	3.1735000	6.3827421	.632
	.10	.00	-6.0389000	6.3827421	.372
		.05	.0517333	6.3827421	.994
		.07	-3.1735000	6.3827421	.632
Cu4	.00	.05	3.7878333*	1.5452665	.040
		.07	4.5588000*	1.5452665	.018
		.10	2.5678000	1.5452665	.135
	.05	.00	-3.7878333*	1.5452665	.040
		.07	.7709667	1.5452665	.631
		.10	-1.2200333	1.5452665	.453
	.07	.00	-4.5588000*	1.5452665	.018
		.05	-.7709667	1.5452665	.631
		.10	-1.9910000	1.5452665	.234
	.10	.00	-2.5678000	1.5452665	.135
		.05	1.2200333	1.5452665	.453
		.07	1.9910000	1.5452665	.234
Mn4	.00	.05	12.3658000	19.9748512	.553
		.07	26.7372333	19.9748512	.218
		.10	17.5735000	19.9748512	.405
	.05	.00	-12.3658000	19.9748512	.553
		.07	14.3714333	19.9748512	.492
		.10	5.2077000	19.9748512	.801
	.07	.00	-26.7372333	19.9748512	.218
		.05	-14.3714333	19.9748512	.492
		.10	-9.1637333	19.9748512	.659
	.10	.00	-17.5735000	19.9748512	.405
		.05	-5.2077000	19.9748512	.801
		.07	9.1637333	19.9748512	.659

Dependent Variable	(I) Condition	(J) Condition	Mean Difference (I-J)	Std. Error	Sig.
Ni4	.00	.05	.4327667	.7987255	.603
		.07	-.6971333	.7987255	.408
		.10	-.9044333	.7987255	.290
	.05	.00	-.4327667	.7987255	.603
		.07	-1.1299000	.7987255	.195
		.10	-1.3372000	.7987255	.133
	.07	.00	.6971333	.7987255	.408
		.05	1.1299000	.7987255	.195
		.10	-.2073000	.7987255	.802
	.10	.00	.9044333	.7987255	.290
		.05	1.3372000	.7987255	.133
		.07	.2073000	.7987255	.802
Pb4	.00	.05	.5353000	.7106038	.473
		.07	.4749000	.7106038	.523
		.10	-.5565000	.7106038	.456
	.05	.00	-.5353000	.7106038	.473
		.07	-.0604000	.7106038	.934
		.10	-1.0918000	.7106038	.163
	.07	.00	-.4749000	.7106038	.523
		.05	.0604000	.7106038	.934
		.10	-1.0314000	.7106038	.185
	.10	.00	.5565000	.7106038	.456
		.05	1.0918000	.7106038	.163
		.07	1.0314000	.7106038	.185
Zn4	.00	.05	4.6001667	4.6286551	.349
		.07	6.9966000	4.6286551	.169
		.10	2.0005000	4.6286551	.677
	.05	.00	-4.6001667	4.6286551	.349
		.07	2.3964333	4.6286551	.619
		.10	-2.5996667	4.6286551	.590
	.07	.00	-6.9966000	4.6286551	.169
		.05	-2.3964333	4.6286551	.619
		.10	-4.9961000	4.6286551	.312
	.10	.00	-2.0005000	4.6286551	.677
		.05	2.5996667	4.6286551	.590
		.07	4.9961000	4.6286551	.312

Appendix F

. reg Co, BCR1

Source	SS	df	MS			
Model	.135216639	2	.06760832	Number of obs =	48	
Residual	.235781587	45	.005239591	F(2, 45) =	12.90	
Total	.370998227	47	.007893579	Prob > F =	0.0000	
				R-squared =	0.3645	
				Adj R-squared =	0.3362	
				Root MSE =	.07239	

col	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]	
time	.0012652	.000961	1.32	0.195	-.0006705	.0032008
om	-.0140829	.0028703	-4.91	0.000	-.0198639	-.0083019
_cons	1.052789	.0228149	46.14	0.000	1.006837	1.09874

. reg Cr, BCR1

Source	SS	df	MS			
Model	.594186076	2	.297093038	Number of obs =	48	
Residual	2.14776932	45	.047728207	F(2, 45) =	6.22	
Total	2.7419554	47	.058339477	Prob > F =	0.0041	
				R-squared =	0.2167	
				Adj R-squared =	0.1819	
				Root MSE =	.21847	

crl	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]	
time	-.0074909	.0029006	-2.58	0.013	-.0133329	-.0016489
om	-.0208263	.0086628	-2.40	0.020	-.0382741	-.0033784
_cons	-.2812894	.0688584	-4.09	0.000	-.4199773	-.1426015

. reg Cu, BCR1

Source	SS	df	MS			
Model	42.2364248	2	21.1182124	Number of obs =	48	
Residual	6.32204413	45	.140489869	F(2, 45) =	150.32	
Total	48.5584689	47	1.03315891	Prob > F =	0.0000	
				R-squared =	0.8698	
				Adj R-squared =	0.8640	
				Root MSE =	.37482	

cul	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]	
time	-.0124266	.0049764	-2.50	0.016	-.0224496	-.0024036
om	-.2550139	.0148626	-17.16	0.000	-.2849486	-.2250791
_cons	1.656986	.1181386	14.03	0.000	1.419043	1.894929


```
. reg Mn, BCRI
```

Source	SS	df	MS	Number of obs =	48
Model	.12680334	2	.06340167	F(2, 45) =	9.73
Residual	.293191524	45	.006515367	Prob > F =	0.0003
				R-squared =	0.3019
				Adj R-squared =	0.2709
Total	.419994865	47	.008936061	Root MSE =	.08072

mn1	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	.0038432	.0010717	3.59	0.001	.0016848 .0060017
om	.0082235	.0032007	2.57	0.014	.001777 .01467
_cons	6.339895	.0254413	249.20	0.000	6.288653 6.391136

```
. reg Ni, BCRI
```

Source	SS	df	MS	Number of obs =	48
Model	.093517701	2	.04675885	F(2, 45) =	0.95
Residual	2.20656138	45	.049034697	Prob > F =	0.3930
				R-squared =	0.0407
				Adj R-squared =	-0.0020
Total	2.30007908	47	.048937853	Root MSE =	.22144

nil	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	-.0004642	.00294	-0.16	0.875	-.0063857 .0054572
om	.0120465	.0087806	1.37	0.177	-.0056385 .0297315
_cons	.6654635	.0697945	9.53	0.000	.5248902 .8060367

```
. reg Pb, BCRI
```

Source	SS	df	MS	Number of obs =	48
Model	10.7381783	2	5.36908915	F(2, 45) =	25.91
Residual	9.32339879	45	.20718664	Prob > F =	0.0000
				R-squared =	0.5353
				Adj R-squared =	0.5146
Total	20.0615771	47	.426842066	Root MSE =	.45518

pbl	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	-.0013383	.0060433	-0.22	0.826	-.0135101 .0108336
om	-.1298767	.018049	-7.20	0.000	-.1662292 -.0935242
_cons	.296179	.1434664	2.06	0.045	.0072229 .5851351

reg Zn, BCR1

Source	SS	df	MS	Number of obs =	48
Model	.354243308	2	.177121654	F(2, 45) =	43.80
Residual	.181957837	45	.004043507	Prob > F =	0.0000
				R-squared =	0.6607
				Adj R-squared =	0.6456
Total	.536201145	47	.011408535	Root MSE =	.06359

zn1	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	.0031852	.0008443	3.77	0.000	.0014848 .0048856
om	-.0215984	.0025215	-8.57	0.000	-.0266768 -.0165199
_cons	2.095627	.0200423	104.56	0.000	2.05526 2.135995

. reg Co, BCR2

Source	SS	df	MS	Number of obs =	48
Model	.181131616	2	.090565808	F(2, 45) =	6.62
Residual	.616046917	45	.013689931	Prob > F =	0.0030
				R-squared =	0.2272
				Adj R-squared =	0.1929
Total	.797178533	47	.016961245	Root MSE =	.117

co2	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	.0004652	.0015534	0.30	0.766	-.0026636 .003594
om	.0168187	.0046395	3.63	0.001	.0074742 .0261631
_cons	1.449244	.0368782	39.30	0.000	1.374968 1.523521

. . reg Cr, BCR2

Source	SS	df	MS	Number of obs =	48
Model	1.84139713	2	.920698567	F(2, 45) =	262.04
Residual	.15811176	45	.003513595	Prob > F =	0.0000
				R-squared =	0.9209
				Adj R-squared =	0.9174
Total	1.99950889	47	.042542742	Root MSE =	.05928

cr2	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	.0159629	.000787	20.28	0.000	.0143778 .017548
om	-.0249478	.0023504	-10.61	0.000	-.0296818 -.0202138
_cons	2.570786	.0186829	137.60	0.000	2.533156 2.608415

. reg Cu, BCR2

Source	SS	df	MS	Number of obs =	48
Model	19.0776459	2	9.53882293	F(2, 45) =	294.48
Residual	1.45766179	45	.032392484	Prob > F =	0.0000
				R-squared =	0.9290
				Adj R-squared =	0.9259
Total	20.5353076	47	.436921439	Root MSE =	.17998

cu2	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	.0075445	.0023895	3.16	0.003	.0027317 .0123573
om	-.1717224	.0071366	-24.06	0.000	-.1860963 -.1573485
_cons	2.302347	.0567272	40.59	0.000	2.188092 2.416601

.. reg Mn, BCR2

Source	SS	df	MS	Number of obs =	48
Model	.211089214	2	.105544607	F(2, 45) =	9.77
Residual	.486016052	45	.010800357	Prob > F =	0.0003
				R-squared =	0.3028
				Adj R-squared =	0.2718
Total	.697105266	47	.014832027	Root MSE =	.10392

mn2	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	.0052886	.0013798	3.83	0.000	.0025096 .0080676
om	-.0090786	.0041209	-2.20	0.033	-.0173785 -.0007787
_cons	6.53553	.0327558	199.52	0.000	6.469556 6.601503

. reg Ni, BCR2

Source	SS	df	MS	Number of obs =	48
Model	.558150921	2	.27907546	F(2, 45) =	32.94
Residual	.381289915	45	.008473109	Prob > F =	0.0000
				R-squared =	0.5941
				Adj R-squared =	0.5761
Total	.939440836	47	.019988103	Root MSE =	.09205

ni2	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	-.0036385	.0012221	-2.98	0.005	-.0061 -.001177
om	.0275592	.00365	7.55	0.000	.0202077 .0349107
_cons	1.429768	.0290129	49.28	0.000	1.371333 1.488203

. reg Pb, BCR2

Source	SS	df	MS			
Model	1.55091567	2	.775457834	Number of obs =	48	
Residual	1.08089364	45	.024019859	F(2, 45) =	32.28	
Total	2.6318093	47	.055995943	Prob > F =	0.0000	
				R-squared =	0.5893	
				Adj R-squared =	0.5710	
				Root MSE =	.15498	

pb2	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]	
time	.015164	.0020577	7.37	0.000	.0110197	.0193084
om	-.0196838	.0061455	-3.20	0.002	-.0320615	-.0073062
_cons	2.945271	.0488489	60.29	0.000	2.846885	3.043658

. reg Zn, BCR2

Source	SS	df	MS			
Model	1.36387329	2	.681936644	Number of obs =	48	
Residual	.385611642	45	.008569148	F(2, 45) =	79.58	
Total	1.74948493	47	.037223084	Prob > F =	0.0000	
				R-squared =	0.7796	
				Adj R-squared =	0.7698	
				Root MSE =	.09257	

zn2	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]	
time	.0133105	.001229	10.83	0.000	.0108351	.0157859
om	.0237516	.0036706	6.47	0.000	.0163586	.0311446
_cons	2.122321	.0291768	72.74	0.000	2.063556	2.181086

. reg Co, BCR3

Source	SS	df	MS			
Model	.833307684	2	.416653842	Number of obs =	48	
Residual	1.98114664	45	.044025481	F(2, 45) =	9.46	
Total	2.81445433	47	.059882007	Prob > F =	0.0004	
				R-squared =	0.2961	
				Adj R-squared =	0.2648	
				Root MSE =	.20982	

co3	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]	
time	-.0037246	.0027858	-1.34	0.188	-.0093354	.0018862
om	.0344455	.00832	4.14	0.000	.0176881	.0512028
_cons	1.826543	.0661335	27.62	0.000	1.693343	1.959742

. reg Cr, BCR3

Source	SS	df	MS	Number of obs =	48
Model	4.59597163	2	2.29798582	F(2, 45) =	54.28
Residual	1.90521318	45	.042338071	Prob > F =	0.0000
				R-squared =	0.7069
				Adj R-squared =	0.6939
Total	6.50118481	47	.138323081	Root MSE =	.20576

cr3	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	-.0044556	.0027319	-1.63	0.110	-.0099579 .0010466
om	.08396	.008159	10.29	0.000	.067527 .1003931
_cons	1.167014	.0648537	17.99	0.000	1.036392 1.297636

. reg Cu, BCR3

Source	SS	df	MS	Number of obs =	48
Model	5.49917813	2	2.74958907	F(2, 45) =	63.64
Residual	1.9443148	45	.043206996	Prob > F =	0.0000
				R-squared =	0.7388
				Adj R-squared =	0.7272
Total	7.44349294	47	.15837219	Root MSE =	.20786

cu3	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	-.0006237	.0027598	-0.23	0.822	-.0061821 .0049348
om	.0929679	.0082423	11.28	0.000	.0763671 .1095688
_cons	1.84279	.0655158	28.13	0.000	1.710834 1.974745

. reg Mn, BCR3

Source	SS	df	MS	Number of obs =	48
Model	5.46392109	2	2.73196054	F(2, 45) =	18.52
Residual	6.63716206	45	.14749249	Prob > F =	0.0000
				R-squared =	0.4515
				Adj R-squared =	0.4271
Total	12.1010831	47	.257469854	Root MSE =	.38405

mn3	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	-.0148663	.0050989	-2.92	0.006	-.0251361 -.0045966
om	.0813617	.0152285	5.34	0.000	.0506899 .1120334
_cons	3.037158	.121047	25.09	0.000	2.793357 3.28096

. reg Ni, BCR3

Source	SS	df	MS	Number of obs =	48
Model	5.64284649	2	2.82142324	F(2, 45) =	30.88
Residual	4.11208656	45	.091379701	Prob > F =	0.0000
				R-squared =	0.5785
				Adj R-squared =	0.5597
Total	9.75493304	47	.207551767	Root MSE =	.30229

ni3	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	-.022506	.0040135	-5.61	0.000	-.0305895 -.0144224
om	.0659875	.0119866	5.51	0.000	.0418453 .0901298
_cons	.5284057	.0952783	5.55	0.000	.3365053 .7203061

. reg Pb, BCR3

Source	SS	df	MS	Number of obs =	48
Model	4.90483078	2	2.45241539	F(2, 45) =	121.22
Residual	.910414731	45	.020231438	Prob > F =	0.0000
				R-squared =	0.8434
				Adj R-squared =	0.8365
Total	5.81524551	47	.123728628	Root MSE =	.14224

pb3	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	-.0143415	.0018885	-7.59	0.000	-.0181451 -.010538
om	.0766641	.0056401	13.59	0.000	.0653044 .0880238
_cons	1.15608	.0448314	25.79	0.000	1.065785 1.246375

. reg Zn, BCR3

Source	SS	df	MS	Number of obs =	48
Model	1.24338984	2	.621694922	F(2, 45) =	11.88
Residual	2.35444429	45	.052320984	Prob > F =	0.0001
				R-squared =	0.3456
				Adj R-squared =	0.3165
Total	3.59783413	47	.076549662	Root MSE =	.22874

zn3	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	.0038254	.0030369	1.26	0.214	-.0022912 .009942
om	.042714	.00907	4.71	0.000	.024446 .060982
_cons	1.542069	.0720953	21.39	0.000	1.396861 1.687276

. reg Co, BCR4

Source	SS	df	MS	Number of obs =	48
Model	.383569749	2	.191784874	F(2, 45) =	28.51
Residual	.30271612	45	.006727025	Prob > F =	0.0000
				R-squared =	0.5589
				Adj R-squared =	0.5393
Total	.686285869	47	.014601827	Root MSE =	.08202

co4	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	.0071018	.0010889	6.52	0.000	.0049086 .0092951
om	-.0123782	.0032522	-3.81	0.000	-.0189285 -.0058278
_cons	1.95713	.0258512	75.71	0.000	1.905063 2.009197

. reg Cr, BCR4

Source	SS	df	MS	Number of obs =	48
Model	.417692947	2	.208846473	F(2, 45) =	38.15
Residual	.246322366	45	.00547383	Prob > F =	0.0000
				R-squared =	0.6290
				Adj R-squared =	0.6126
Total	.664015313	47	.014127985	Root MSE =	.07399

cr4	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	.0085755	.0009823	8.73	0.000	.0065971 .010554
om	-.0008871	.0029337	-0.30	0.764	-.0067959 .0050217
_cons	4.450428	.0233193	190.85	0.000	4.40346 4.497395

. reg Cu, BCR4

Source	SS	df	MS	Number of obs =	48
Model	1.63690619	2	.818453093	F(2, 45) =	42.67
Residual	.863143989	45	.019180978	Prob > F =	0.0000
				R-squared =	0.6547
				Adj R-squared =	0.6394
Total	2.50005017	47	.053192557	Root MSE =	.1385

cu4	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	-.0166915	.0018388	-9.08	0.000	-.020395 -.012988
om	-.0094145	.0054917	-1.71	0.093	-.0204754 .0016463
_cons	3.982373	.043652	91.23	0.000	3.894454 4.070293

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. reg Mn, BCR4
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Source	SS	df	MS	Number of obs =	48
Model	.058913474	2	.029456737	F(2, 45) =	1.38
Residual	.961963951	45	.021376977	Prob > F =	0.2625
Total	1.02087742	47	.021720796	R-squared =	0.0577
				Adj R-squared =	0.0158
				Root MSE =	.14621

mn4	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	-.0018191	.0019412	-0.94	0.354	-.0057288 .0020907
om	-.0079445	.0057976	-1.37	0.177	-.0196214 .0037324
_cons	6.016562	.0460832	130.56	0.000	5.923746 6.109379

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. reg Ni, BCR4
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Source	SS	df	MS	Number of obs =	48
Model	.095709841	2	.04785492	F(2, 45) =	1.44
Residual	1.49824708	45	.03329438	Prob > F =	0.2483
Total	1.59395692	47	.033913977	R-squared =	0.0600
				Adj R-squared =	0.0183
				Root MSE =	.18247

ni4	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	-.003971	.0024226	-1.64	0.108	-.0088503 .0009084
om	.0031359	.0072353	0.43	0.667	-.0114367 .0177086
_cons	2.310835	.0575115	40.18	0.000	2.195001 2.426669

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. reg Pb, BCR4
```

Source	SS	df	MS	Number of obs =	48
Model	.06672509	2	.033362545	F(2, 45) =	0.06
Residual	25.8627813	45	.574728473	Prob > F =	0.9437
Total	25.9295064	47	.551691625	R-squared =	0.0026
				Adj R-squared =	-0.0418
				Root MSE =	.75811

pb4	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	.0009137	.0100653	0.09	0.928	-.0193588 .0211862
om	.0098725	.030061	0.33	0.744	-.0506733 .0704184
_cons	2.860025	.2389464	11.97	0.000	2.378762 3.341288


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. reg Zn, BCR4
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Source	SS	df	MS	Number of obs =	48
Model	.060442642	2	.030221321	F(2, 45) =	1.97
Residual	.69177353	45	.015372745	Prob > F =	0.1519
				R-squared =	0.0804
				Adj R-squared =	0.0395
Total	.752216173	47	.016004599	Root MSE =	.12399

zn4	Coef.	Std. Err.	t	P> t	[95% Conf. Interval]
time	-.002616	.0016461	-1.59	0.119	-.0059315 .0006995
om	-.0058303	.0049164	-1.19	0.242	-.0157325 .0040718
_cons	4.286212	.0390791	109.68	0.000	4.207503 4.364922

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