


PREPARATION AND CHARACTERISATION OF  
POLY (VINYL ALCOHOL)/CHITOSAN/SILICON DIOXIDE (PVA/CS/SiO<sub>2</sub>)  
BEADS FOR THE CHROMIUM (VI) (Cr (VI)) REMOVAL

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*Especially dedicated to the beloved one:*

*For my Abah and Ma,  
Haji Sulaiman bin Dollah and Hajah Noriza binti Yusof,  
There's no love and sacrifices like yours,  
And nobody can replace both of you,  
And yet not even a single word can be expressed to thank all of your kindness  
towards me,*

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With Allah's will, your love and supports make me strong and move forwards,  
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Remain the same forever...*



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## ABSTRACT

Chromium pollution has become a global issue and many techniques have been developed for the removal of Chromium (VI) (Cr(VI)) from wastewater. In this study, blend polymer absorbent beads which was poly (vinyl alcohol)/chitosan/silicon dioxide (PVA/CS/SiO<sub>2</sub>) beads were prepared by dropwise method by crosslinking with sodium hydroxide and ethanol and further crosslinked with glutaraldehyde for the purpose of Cr(VI) ion removal in aqueous solution under UV irradiation. All of the samples of PVA/CS/SiO<sub>2</sub> beads were put under UV irradiation for two hours in the high acidic medium of Cr(VI) solution. After that, the Cr solution was analysed by atomic absorption spectroscopy (AAS), and the beads have been characterised by scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDX) and fourier transform infrared spectroscopy (FTIR) analysis. pH value for each sample before and after the absorption test also has been measured. As a result, the data obtained by AAS showed that the Cr(VI) solution of PVA/CS/SiO<sub>2</sub> beads of PCS 25 recorded the most percentage of Cr(VI) absorption which was 99.9% meanwhile PCS 1 recorded the less percentage of Cr(VI) absorption which was 93.4%. Beads morphology images showed that all of the 25 samples of PVA/CS/SiO<sub>2</sub> beads revealed macro and micro pores on the surface of beads before the Cr(VI) absorption and the pore size increased with the increment of CS and SiO<sub>2</sub> concentration. The pores for all of the sample beads before the Cr absorption can be clearly seen. However, the surface of beads became smooth after Cr(VI) absorption, indicating that the pores on the surface were filled up with Cr. This change was further supported by EDX analysis which provided the existence of Cr peaks as the direct evidence. Whereas, FTIR results showed small shifts in wavelength at several peaks before and after the Cr absorption for each sample indicated that there was metal binding process taking place at the surface of the beads. The results of pH value of Cr after the absorption test showed that PCS 1 recorded the lowest pH value which was 5.81 and PCS 25 recorded the highest pH value which was 6.08. As for the conclusion, PCS 25 has been chosen as the best absorbent with the highest Cr absorption and the results suggested that, the use of PVA/CS/SiO<sub>2</sub> beads is a sustainable strategy for eliminating Cr(VI) from industrial wastewater.

## ABSTRAK

Pencemaran kromium telah menjadi isu global dan banyak kaedah telah dimajukan untuk penyingkiran kromium (VI) (Kr(VI)) dari air buangan. Dalam kajian ini, manik penyerap polimer kisaran iaitu manik polivinil alkohol/citosan/silikon dioksida (PVA/CS/SiO<sub>2</sub>) telah dihasilkan melalui kaedah bertitisan dengan sambungan silang oleh natrium hidroksida dengan etanol dan sambungan silang selanjutnya dengan glutaraldehid bagi tujuan penyingkiran ion Kr(VI) di dalam larutan akuas di bawah sinaran UV. Kesemua sampel manik PVA/CS/SiO<sub>2</sub> diletakkan di bawah sinaran UV selama dua jam di dalam larutan Kr(VI) yang bermedium asid tinggi. Selepas itu, larutan Kr telah dianalisis oleh kespktroskopan penyerapan atom (AAS), manakala manik PVA/CS/SiO<sub>2</sub> telah diperincikan dengan miroskopi electron penskanan (SEM), spektroskopi penyebaran tenaga x-ray (EDX) dan spektroskopi inframerah transformasi fourier (FTIR). Nilai pH untuk kesemua sampel sebelum dan selepas ujikaji penyerapan juga telah diukur. Hasilnya, data yang telah diperolehi melalui AAS telah menunjukkan bahawa larutan Kr(VI) oleh manik PVA/CS/SiO<sub>2</sub> untuk PCS 25 telah mencatatkan peratusan penyerapan Kr(VI) yang tertinggi dengan 99.9%, manakala PCS 1 mencatatkan peratusan penyerapan Kr(VI) yang terendah dengan 93.4%. Imej morfologi pula telah menunjukkan kesemua 25 sampel manik PVA/CS/SiO<sub>2</sub> mendedahkan liang-liang makro dan mikro pada permukaan manik PVA/CS/SiO<sub>2</sub> sebelum penyerapan Kr(VI) dan saiz liang bertambah dengan pertambahan kepekatan CS dan SiO<sub>2</sub>. Liang-liang untuk kesemua sampel manik sebelum penyerapan Kr boleh dilihat dengan jelas. Namun begitu, permukaan manik ini kelihatan licin selepas penyerapan Kr(VI), menunjukkan bahawa permukaan ini telah terisi dengan Kr. Perubahan ini telah dikukuhkan lagi dengan analisis EDX yang menunjukkan kewujudan puncak Kr sebagai bukti. Sementara itu, hasil keputusan FTIR pula telah menunjukkan beberapa anjakan kecil pada panjang gelombang di beberapa puncak sebelum dan selepas penyerapan Kr bagi setiap sampel menunjukkan bahawa terdapat proses pengikatan logam yang mengambil tempat pada permukaan manik. Hasil keputusan nilai pH pada Kr selepas ujikaji penyerapan pula telah menunjukkan PCS 1 mencatatkan nilai pH yang terendah iaitu 5.81 manakala PCS 25 pula mencatatkan nilai pH yang tertinggi iaitu 6.08. Kesimpulannya, PCS 25 telah dipilih sebagai penyerap terbaik dengan penyerapan Kr yang tertinggi dan hasil kajian mencadangkan bahawa penggunaan manik PVA/CS/SiO<sub>2</sub> adalah satu langkah lestari untuk menyingkirkan Kr(VI) daripada air buangan industri.

## CONTENTS

<b>TITLE</b>		<b>i</b>
<b>DECLARATION</b>		<b>ii</b>
<b>DEDICATION</b>		<b>iii</b>
<b>ACKNOWLEDGEMENT</b>		<b>iv</b>
<b>ABSTRACT</b>		<b>v</b>
<b>ABSTRAK</b>		<b>vi</b>
<b>TABLE OF CONTENTS</b>		<b>vii</b>
<b>LIST OF TABLES</b>		<b>xii</b>
<b>LIST OF FIGURES</b>		<b>xvi</b>
<b>LIST OF ABBREVIATIONS</b>		<b>xxx</b>
<b>LIST OF APPENDICES</b>		<b>xxxiii</b>
<b>CHAPTER 1</b>	<b>INTRODUCTION</b>	<b>1</b>
	1.1 Research background	1
	1.2 Problem statement	2
	1.3 Objectives	4
	1.4 Scope of study	4
	1.5 Significance of study	4
<b>CHAPTER 2</b>	<b>LITERATURE REVIEW</b>	<b>6</b>
	2.1 Heavy metal pollution	6
	2.1.1 Chromium pollution	7
	2.1.2 Water treatment method for heavy metals	9



2.2	Beads as absorbent for wastewater treatment	10
2.2.1	Types of absorbent	11
2.2.1.1	Ceramic beads	12
2.2.1.2	Ceramic beads fabrication process	14
2.2.1.3	Polymer beads	18
2.2.1.4	Polymer beads fabrication process	20
2.3	Polymer beads as absorbent for wastewater treatment	24
2.3.1	Poly (vinyl alcohol) (PVA)	24
2.3.1.1	Crosslink process of PVA	25
2.3.1.2	Fabrication of PVA beads	26
2.3.1.3	Structure and properties	27
2.3.2	Chitosan	29
2.3.2.1	Crosslink process of Chitosan	30
2.3.2.2	Fabrication of Chitosan beads	32
2.3.2.3	Structure and properties	34
2.3.3	PVA/Chitosan beads	37
2.3.3.1	Fabrication of PVA/Chitosan beads	42
2.3.3.2	Roles of PVA/Chitosan beads for heavy metal removal	44
2.3.4	Additives in polymer beads	45
2.3.4.1	Silicon dioxide (SiO <sub>2</sub> ) as additive	46
2.4	Factors affecting heavy metal absorption process	48
2.4.1	Influence of pH	48
2.4.2	Influence of temperature	49



2.4.3	Influence of concentration of heavy metals	50
2.4.4	Influence of different types of light illumination	51
2.5	Chapter summary	53
<b>CHAPTER 3</b>	<b>METHODOLOGY</b>	<b>54</b>
3.1	Introduction	54
3.2	Raw materials	56
3.3	Procedure	56
3.3.1	Preparation of SiO <sub>2</sub> from rice husk	57
3.3.2	Preparation of Chitosan and SiO <sub>2</sub> at different concentration	58
3.4	Sample designation	58
3.5	PVA/CS/SiO <sub>2</sub> beads fabrication	59
3.6	Absorption experiment	60
3.7	Beads characterisations	62
3.7.1	Scanning electron microscope (SEM) analysis	62
3.7.2	Energy dispersive x-ray Spectroscopy (EDX) analysis	63
3.7.3	Fourier transform infrared spectroscopy (FTIR) analysis	64
3.7.4	Atomic absorption spectroscopy (AAS) analysis	66
3.7.5	X-ray diffractometer (XRD) analysis	67
<b>CHAPTER 4</b>	<b>RESULTS AND DISCUSSIONS</b>	<b>69</b>
4.1	Determination of the ideal composition raw materials for beads fabrication	69
4.1.1.	The ideal composition of raw	69



	materials, solvent for PVA and CS, and the crosslinking agent	
4.1.2	The ideal solvent for PVA and CS	73
4.1.3	The ideal types and composition of crosslinking agent	76
4.1.4	The ideal composition for all materials and chemical reagent for the PVA/CS/SiO <sub>2</sub> beads fabrication	81
4.2	Characterisation of SiO <sub>2</sub> by x-ray diffractometer (XRD) analysis	82
4.3	Surface morphology study of PVA/CS/SiO <sub>2</sub> beads by SEM	84
4.4	Characterisation of PVA/CS/SiO <sub>2</sub> beads	86
4.4.1	Elemental analysis of PVA/CS/SiO <sub>2</sub> beads by EDX	86
4.4.2	Molecular bonding analysis of PVA/CS/SiO <sub>2</sub> beads by FTIR analysis	88
4.5	Visual and physical observation in terms of absorption activity onto the PVA/CS/SiO <sub>2</sub> beads	91
4.5.1	Visual observation of PVA/CS/SiO <sub>2</sub> beads upon Cr(VI) sorption	91
4.5.2	pH measurement for each sample after the absorption test	93
4.6	Absorption capability	94
4.6.1	Atomic absorption spectroscopy (AAS) analysis	94
4.6.2	Effect of CS concentration onto PVA/CS/SiO <sub>2</sub> beads on absorption capability of Cr(VI) ion	96
4.6.3	Effect of SiO <sub>2</sub> concentration onto PVA/CS/SiO <sub>2</sub> beads on	97



absorption capability of Cr(VI) ion

<b>CHAPTER 5</b>	<b>CONCLUSIONS AND RECOMMENDATIONS</b>	<b>100</b>
5.1	Conclusion	100
5.2	Recommendations	101
<b>REFERENCES</b>		<b>102</b>
<b>APPENDIX A</b>		<b>117</b>
<b>APPENDIX B</b>		<b>123</b>
<b>APPENDIX C</b>		<b>148</b>
<b>APPENDIX D</b>		<b>173</b>



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**LIST OF TABLES**

2.1	Types of heavy metals, the effect of heavy metals towards human health and the maximum concentration limit (MCL) for drinking water	6
2.2	Comparison between the general chemical characteristics of Cr(VI) and Cr(III)	7
2.3	Types of harmful/diseases caused by Cr(VI)	8
2.4	Four main methods of porous glass fabrication	13
2.5	Types of beads, the materials used for the beads fabrication and the applicable methods	15
2.6	Preparation of porous glass beads by subcritical water treatment method	16
2.7	Hydroxylation process of prepared porous glass beads	17
2.8	The process of CS coating on porous glass beads	17
2.9	Specific surface area, pore volume and mean pore size of porous glass beads supported with CS	18
2.10	Polymer beads and its function towards heavy metals from water	19



2.11	Modified Chitosan beads and their crosslinking process	20
2.12	Types of polymer beads and its methods of fabrication	22
2.13	PVA and its crosslinking agent	25
2.14	Details preparation of PVA beads	26
2.15	PVA properties	28
2.16	CS and its crosslinking agent	31
2.17	Various preparation of CS beads	33
2.18	CS properties	35
2.19	Summarised FTIR analysis based on spectra shown in Fig. 2.17	40
2.20	Summarised FTIR analysis based on Fig. 2.18	41
2.21	Details of fabrication of PVA/CS beads	42
2.22	Additives and its application in polymeric beads	46
2.23	Types of heavy metals used and their optimum pH	48
2.24	Percentage rate of reduction of Cr(VI), light intensity and wavelength of different types of illumination	52
3.1	Process of SiO <sub>2</sub> preparation from rice husk	57
3.2	Different concentration of Chitosan with its suitable volume of acetic acid	58



3.3	Different concentration of SiO <sub>2</sub>	58
3.4	Samples of PVA/CS/SiO <sub>2</sub> beads fabricated for this study	58
4.1	The effect of using small amount of PVA	69
4.2	The effect of using large amount of PVA and the resulting beads	70
4.3	The effect of CS composition onto its swelling behavior	72
4.4	Types of acid used and its dissolving rate	74
4.5	The results of using various concentration of acetic acid	75
4.6	The results of using different concentration of NaOH	78
4.7	Different shape of PVA/CS/SiO <sub>2</sub> beads in three different types of crosslinking agent	79
4.8	The effect of using different concentrations of glutaraldehyde	80
4.9	The ideal composition of raw materials and solvent	82
4.10	The ideal types and compositions of crosslinking agents	82
4.11	SiO <sub>2</sub> phases detected in fired rice husk with ICDD file number and its major peaks	83
4.12	Characteristic of SiO <sub>2</sub> at the temperature of 800°C	83
4.13	EDX analysis on the surface of beads (PCS 25)	87



- before and after the Cr absorption
- 4.14 Wavenumber and types of bonding for PVA/CS/SiO<sub>2</sub> beads (PCS 25) before and after CR absorption performed by FTIR analysis 89
- 4.15 Comparison of FTIR spectra analysis before absorption of heavy metals between the present works with Hui *et al.*, (2014a), Gandhi *et al.*, (2011), Li & Bai, 2005, Wu *et al.*, (2010) and Cani & Pescarmona, (2014). 89
- 4.16 pH of Cr(VI) solution for each sample after the absorption test 93
- 4.17 Results of Cr(VI) concentration for each sample of PVA/CS/SiO<sub>2</sub> beads performed by AAS analysis 94
- 4.18 Percentage (%) absorption of Cr(VI) for each sample of beads 95



**LIST OF FIGURES**

2.1	CS–cellulose hydrogel beads	11
2.2	Optical images of BG gels template by (a) P123 and (b) F108 after dropping into $\text{CHCl}_3$ . Microscope images of BG beads (c) before and (d) after calcination at $600^\circ\text{C}$	14
2.3	FE-SEM images of calcined BG beads. (a) Low magnification images of beads and (b) and (c) high magnification images of surface of beads	15
2.4	Steps of porous glass beads fabrication	15
2.5	(a and b): Morphology of porous glass beads treated with hydrochloric acid; (c and d): morphology of porous glass beads supported with CS	18
2.6	General steps of polymeric beads fabrication process	21
2.7	Chemical reaction of PVA with boric acid	26
2.8	SEM images of the PVA hydrogel beads (a) before absorption of phosphate and (b) after absorption of phosphate	28



2.9	FTIR spectra of the PVA hydrogel beads (a) before absorption of phosphate and (b) after absorption of phosphate	29
2.10	SEM images show the surface morphology of CS-cellulose and crosslinked CS-cellulose hydrogel beads: (a) CS-cellulose and (b) crosslinked CS-cellulose	32
2.11	Structure of CS	34
2.12	SEM images of (a) GCB and (b) chromium sorbed GCB	35
2.13	FTIR spectra of (a) GCB and (b) Cr sorbed GCB	36
2.14	Proposed structure of blend CS/PVA	37
2.15	(a) The shape of PVA/CS beads, and (b and c) porous morphologies of PVA/CS beads by SEM	38
2.16	SEM of modified PVA/CS beads: (a) before activation and (b) activated by glutaraldehyde	39
2.17	FTIR spectra of (A) crosslinked PVA/CS beads and (B) crosslinked PVA/CS beads after absorption of Cd(II)	39
2.18	FTIR spectra of the unloaded PVA/CS hydrogel absorbent (a) and samples after Hg(II) absorption in different pH solutions (b: pH 2.00; c: pH 5.00; d: pH 7.00; e: pH 9.00; f: pH 11.00)	41



2.19	Effect of contact time on the adsorption of $\text{Cu}^{2+}$ , $\text{Pb}^{2+}$ , $\text{Zn}^{2+}$ and $\text{Cd}^{2+}$ onto PVA/CS beads	50
2.20	Effect of initial concentration on the removal efficiency of Cr(VI)	51
2.21	Influence of different types of illumination on the reduction of Cr(VI) at initial concentration of 50 mg/L; pH 1; catalyst dosage 8% (v/v)	52
3.1	Flow chart of PVA/CS/SiO <sub>2</sub> beads preparation and its characterisation	55
3.2	Flow chart of PVA/CS/SiO <sub>2</sub> beads fabrication	59
3.3	JSM 6380 LA- SEM-EDX	63
3.4	JFC-1800 Auto Fine Coater	64
3.5	Spectrum 100 FTIR spectroscopy	65
3.6	Atomic absorption spectroscopy by using flame technique	66
3.7	Bruker D8 Advance x-ray diffractometer	67
3.8	The sample preparation for rice husk ash onto the powder holder	68
3.9	Powder diffractometer	68
4.1	Uniform spherical shaped beads	71
4.2	PVA/CS/SiO <sub>2</sub> solution a) without mechanical stirring	73



PT T A U T H M  
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- 4.3 CS mixture dissolved in nitric acid yielded the agglomerate beads 74
- 4.4 A gel-like solution of CS with acetic acid after homogeneously stirred 75
- 4.5 The agglomerate of PVA/CS/SiO<sub>2</sub> stick to each other when crosslinked in 0.5 M of boric acid at a) top view and b) front view 77
- 4.6 PVA/CS/SiO<sub>2</sub> beads in (a) boric acid (1.0 M, 100 mL), (b) NaOH (1.0 M, 100 mL) + ethanol (25 mL) and (c) NaOH (5.0 M, 100 mL) + ethanol (25 mL) 79
- 4.7 The swelling beads (without glutaraldehyde) after Cr(VI) absorption test after (a) 30 minutes and (b) 1 hour 81
- 4.8 Amorphous SiO<sub>2</sub> spectrum obtained by XRD analysis 83
- 4.9 Representative surface morphology of PVA/CS/SiO<sub>2</sub> beads (PCS 25) at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) 84
- 4.10 Representative EDX spectrum of PVA/CS/SiO<sub>2</sub> beads (PCS 25) before Cr(VI) absorption 86
- 4.11 Representative EDX spectrum of PVA/CS/SiO<sub>2</sub> beads (PCS 25) after Cr(VI) absorption 86



4.12	Quantitative result of EDX for PVA/CS/SiO <sub>2</sub> beads (PCS 25)	88
4.13	Representative FTIR spectrum of PVA/CS/SiO <sub>2</sub> beads for PCS 25 before and after Cr absorption	88
4.14	Representative FTIR spectrum of PVA/CS/SiO <sub>2</sub> beads for PCS 25 after the Cr absorption	90
4.15	Chromium solution before and after absorption of PVA/CS/SiO <sub>2</sub> beads (PCS 25)	92
4.16	PVA/CS/SiO <sub>2</sub> beads (PCS 25) before and after the Cr absorption	92
4.17	Graph of effect of different CS addition on Cr(VI) absorption	96
4.18	Graph of effect of different SiO <sub>2</sub> addition on Cr(VI) absorption	98
4.19	Surface morphology of PVA/Chitosan/SiO <sub>2</sub> beads PCS 1 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption)	Apndx. B
4.20	Surface morphology of PVA/Chitosan/SiO <sub>2</sub> beads PCS 2 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption)	Apndx. B
4.21	Surface morphology of PVA/Chitosan/SiO <sub>2</sub> beads PCS 3 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and	Apndx. B



- c) 1000 (after Cr(VI) absorption)
- 4.22 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 4 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.23 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 5 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.24 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 6 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.25 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 7 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.26 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 8 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.27 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 9 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.28 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 10 at magnification of a) 25 (the whole



bead) b) 1000 (before Cr(VI) absorption) and  
c) 1000 (after Cr(VI) absorption)

- 4.29 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 11 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.30 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 12 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.31 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 13 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.32 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 14 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.33 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 15 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.34 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 16 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.35 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads Apndx. B



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PCS 17 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption)

4.36 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads Apndx. B  
PCS 18 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption)

4.37 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads Apndx. B  
PCS 19 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption)

4.38 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads Apndx. B  
PCS 20 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption)

4.39 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads Apndx. B  
PCS 21 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption)

4.40 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads Apndx. B  
PCS 22 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption)

4.41 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads Apndx. B  
PCS 23 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption)



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- 4.42 Surface morphology of PVA/Chitosan/SiO<sub>2</sub> beads PCS 24 at magnification of a) 25 (the whole bead) b) 1000 (before Cr(VI) absorption) and c) 1000 (after Cr(VI) absorption) Apndx. B
- 4.43 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 1) (a) before and (b) after Cr(VI) absorption performed by EDX Apndx. C
- 4.44 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 2) (a) before and (b) after Cr(VI) absorption performed by EDX Apndx. C
- 4.45 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 3) (a) before and (b) after Cr(VI) absorption performed by EDX Apndx. C
- 4.46 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 4) (a) before and (b) after Cr(VI) absorption performed by EDX Apndx. C
- 4.47 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 5) (a) before and (b) after Cr(VI) absorption performed by EDX Apndx. C
- 4.48 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 6) (a) before and (b) after Cr(VI) absorption performed by EDX Apndx. C
- 4.49 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 7) (a) before and (b) after Cr(VI) absorption performed by EDX Apndx. C
- 4.50 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads Apndx. C



- (PCS 8) (a) before and (b) after Cr(VI)  
absorption performed by EDX
- 4.51 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 9) (a) before and (b) after Cr(VI)  
absorption performed by EDX Apndx. C
- 4.52 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 10) (a) before and (b) after Cr(VI)  
absorption performed by EDX Apndx. C
- 4.53 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 11) (a) before and (b) after Cr(VI)  
absorption performed by EDX Apndx. C
- 4.54 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 12) (a) before and (b) after Cr(VI)  
absorption performed by EDX Apndx. C
- 4.55 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 13) (a) before and (b) after Cr(VI)  
absorption performed by EDX Apndx. C
- 4.56 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 14) (a) before and (b) after Cr(VI)  
absorption performed by EDX Apndx. C
- 4.57 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 15) (a) before and (b) after Cr(VI)  
absorption performed by EDX Apndx. C
- 4.58 Spectra images of PVA/chitosan/SiO<sub>2</sub> beads (PCS 16) (a) before and (b) after Cr(VI)  
absorption performed by EDX Apndx. C



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