UNIVERSITY OF ZIMBABWE



AVOCADO SEED STARCH AND CARBOXYMETHYL CELLULOSE SUPERABSORBENT POLYMER: SYNTHESIS AND CHARACTERISATION

KETIWE SIYADUBA

REGISTRATION NUMBER: R 073944L

A DISSERTATION SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE MASTER OF SCIENCE DEGREE IN CHEMISTRY

2016

FACULTY OF SCIENCE

CHEMISTRY DEPARTMENT

SUPERVISORS: DR. L. NHAMO, DR. S. BEBE, PROF. R. MAFOTI

DECLARATION

I, KETIWE SIYADUBA, do hereby declare that this dissertation is a result of my own investigation and research, except to the extent indicated in the Acknowledgements, References and comments included in the body of the report, and that it has not been submitted in part or in full for any other degree to any other university.

••••••

Student Signature

••••••

Supervisor Signature

.....

Date

Date

ACKNOWLEDGEMENTS

Firstly I would like to thank God for granting me the knowledge and the capacity to learn throughout the Master of Science in Chemistry Degree programme at the University of Zimbabwe. I would like to extend my gratitude to my family and friends who were patient and assisted me as I undertook my studies.

I wish to thank Dr Nhamo, Dr Bebe and Professor Mafoti for the scientific guidance and constructive advice that steered this research project to completion.

I would like to thank the Scientific and Industrial Research and Development Centre staff particularly the Food and Biomedical Technology Institute Scientists and Interns for their support and assistance in carrying out the experimental work.

Abstract

Superabsorbent polymers (SAPs) of crosslinked avocado seed starch and carboxymethyl cellulose were synthesized and their performance as fluid absorption materials was assessed by comparing them with those made from analytical grade starch. The SAPs were characterized by water absorption capacity (WAC) tests, Fourier transform infrared spectroscopy analyses, solubility fraction tests, effects of crosslinker quantity and swelling rate were investigated to determine the suitability of avocado starch in the synthesis of SAPs.

Avocado seed starch was used in the superabsorbent polymer framework because of its biodegradability, abundance and limited use as a source of food for human consumption. The results of the research showed that the SAPs produced with avocado seed starch and those made from pure starch have comparable properties. The WAC for the avocado seed starch blend was 39g/g (3900% of the original weight) when the ratio of the avocado starch, carboxymethyl cellulose and aluminium sulphate was 88: 9.7: 2.3 respectively. The pure starch blend with a similar weight ratio had a WAC of 46.5g/g. As the amount of either starch was increased the differences between the WAC of the SAPs were less than 3.0g/g. FTIR spectra of the SAPs showed no significant differences between avocado starch and pure starch SAPs. The soluble fraction results showed that avocado SAPs had good gel formation. Analysis of experimental data by GraphPad Prism 6.0, one way Anova showed that there was no significant difference between the two types of SAPs for all the tests conducted.

Avocado seed starch can therefore be used in the synthesis of superabsorbent polymers. It can be used as a substitute for starch extracted from agricultural plants that have already been studied in detail.

Key words: Carboxymethyl cellulose, starch, superabsorbent polymer, water absorption capacity, biodegradable

TABLE OF CONTENTS

Declaration	.ii
Acknowledgements	iii
Abstract	iv
ist of tables	X
ist of figures	xi

СНАР	TER 1	1
1.0 IN'	TRODUCTION	1
1.1 Ba	ckground of study	1
1.2 Pro	blem statement	5
1.3 Air	n	5
1.4 Spe	ecific objectives	6
1.5 Res	search questions	6
1.6 Res	search proposition	7
1.7 De	limitation of study	7
1.8 Lin	nitations	7
1.9 Jus	tification of the research	8
1.10	Literature review	.9
1.10.1	Definitions	9
1.10.2	History of superabsorbent polymers	10
1.10.3	Synthesis of biodegradable superabsorbent polymers	11
1.10.4	Chemistry of CMC, avocado starch and aluminium sulphate	13
1.10.5	Starch	14

1.10.6 Avocado seed starch	16
1.10.7 Carboxymethyl cellulose	17
1.10.8 Aluminium sulphate octadeccahydrate	20
1.10.9 Crosslinking in SAPs	20
1.10.10 Classification of SAPs	23
1.10.11 Characterisation of superabsorbent polymers	.25
1.10.12 Water absorption capacity	27
1.10.13 Effects of salinity	29
1.10.14 Absorbency under load (AUL)	30
1.10.15 Wicking capacity and rate	.30
1.10.16 Swelling rate	31
1.10.17 Soluble fraction	.31
1.10.18 Ionic sensitivity	.32
1.10.19 Other properties of super-absorbing materials	.32
1.10.20 Proposed crosslinking model	33
1.10.21 Applications of SAPs	36

СНАР	TER 2	38
2.0 RE	SEARCH METHODOLOGY	38
2.1 Int	roduction	38
2.2 Ma	terials and procedures	38
2.2.1	Materials	38
2.2.2	Equipment	38
2.2.3	Statistical evaluation of experimental results	39

2.3 Sy	nthesis of the avocado starch based superabsorbent polymer	39
2.4 Iso	lation of starch from avocado seeds	39
2.4.1	Determination of starch content in the extracted avocado seed starch	41
2.4.2	Elemental analysis of the avocado seed powder and the starch	42
2.4.3	Determination of the ratio of seed to fruit	42
2.5 Sy	nthesis of the avocado seed superabsorbent polymer (SAP)	42
2.6 Inv	vestigation of optimal crosslinking of avocado starch and CMC	45
2.7 Ch	aracterisation of the superabsorbent polymers	46
2.7.1	Structural analysis	46
2.7.2	Water absorption capacity (WAC)	47
2.7.3	Soluble fraction	48
2.7.4	Ability to rehydrate	49

CHAPTER 3	0
3.0 RESULTS AND DISCUSSION	0
3.1 Introduction	0
3.2 Extraction of starch from the avocado seed	0
3.2.1 Results for the analysis of starch content of avocado seed starch	1
3.3 Results of mineral analysis of avocado starch and dried avocado powder	7
3.4 Synthesized avocado seed starch superabsorbent polymer	8
3.5 Results for tests of the synthesized superabsorbent polymers	9
3.5.1 Fourier transform infrared spectroscopy analysis results5	9

3.5.2 Water absorption capacity	63
3.5.3 Soluble fraction	69
3.5.4 Ability to rehydrate	
3.5.5 Statistical analysis	70
3.6 Discussion summary	72
3.6.1 Starting materials	73
3.6.2 Synthesis of the superabsorbent polymer	
3.6.3 Water absorption capacity of synthesized SAPs	74
3.6.4 Soluble fraction	

CHAPTER 4	.76
4.0 Conclusion	76
4.1 Introduction	76
4.2 Conclusions	76
4.3 Test of the research proposition	77
4.4 Recommendations	78

REFERENCES	79

Appendix 1 A comparison of pure starch SAPs against avocado seed starch SAPs..... 88

Appendix 2 Comparison of WAC of starch SAP acocado starch SAP with varying aluminium	
sulphate	89
Appendix 3 Soluble fraction of SAPs	90

LIST OF TABLES	PAGE
Table 2.0: Glucose standards	41

Table 2.1: Material compositions for SAF	synthesis	44
1		

Table 2.2: Investigation of optimal crosslinking for high water absorpt	ion capacity
(WAC)	45
Table 3.1: Concentration of avocado starch	56
Table 3.2: Analysis of mineral content	57
Table 3.3: Compositions of SAP analysed by FTIR	61
Table 3.4: GraphPad Prism 6.0 statistical output	71

LIST OF FIGURES

Figure1.0: Applications of SAP2	
Figure 1.1: Hydrophilic functional groups for the polymer backbone	
Figure 1.2: Molecular structure of starch (Lu et al., 2009)15	
Figure 1.3: FTIR spectrum of native starch16	
Figure 1.4: Chemical structure of CMC	
Figure 1.5:FTIR spectrum of CMC 19	
Figure 1.6: Optimisation of water retention of potato starch hydrogel at various levels of	
crosslinking23	
Figure 1.7: Typical AUL testing apparatus	
Figure 1.8: Proposed crosslinking model for Al ion and CMC	
Figure 1.9: Proposed crosslinking model for Al ion /CMC/starch blend	
Figure 1.10: FTIR spectrum for CMC, starch and CMC/starch blend	
Figure 2.0: Procedure for the extraction of starch	
Figure 3.0: The extraction of starch from avocado seeds	
Figure 3.1: Calibration curve for glucose standards	
Figure 3.2: Starch FTIR spectrum	
Figure 3.3: FTIR spectrum for avocado seed starch 55	
Figure 3.4: Starch and Avocado seed starch FTIR spectra	
Figure 3.5: Synthesized starch and avocado seed starch SAP	

Figure 3.6: Superimposed	spectra of CMC, star	rch, avocado starch, a	aluminium sulphate and

synthesized SAPs	60	
Figure 3.7: A comparison of CMC SAP, starch/CMC composites and avocado		
starch/CMC	62	
Figure 3.8: Water absorption of avocado starch and starch SAP	65	
Figure 3.9: A comparison of pure starch SAPs against avocado seed starch SAPs.	66	
Figure 3.10: A comparison of the WAC of pure starch SAPs against avocado	seed	starch
SAPs	67	
Figure 3.11: The effect of aluminium sulphate concentration on WAC of SAPs	68	
Figure 3.12: Soluble fraction of avocado and starch SAPs	69	