



شبكة المعلومات الجامعية
التوثيق الإلكتروني والميكروفيلم

بسم الله الرحمن الرحيم



MONA MAGHRABY



شبكة المعلومات الجامعية
التوثيق الإلكتروني والميكرو فيلم



شبكة المعلومات الجامعية التوثيق الإلكتروني والميكرو فيلم



MONA MAGHRABY



شبكة المعلومات الجامعية
التوثيق الإلكتروني والميكروفيلم

جامعة عين شمس

التوثيق الإلكتروني والميكروفيلم

قسم

نقسم بالله العظيم أن المادة التي تم توثيقها وتسجيلها
علي هذه الأقراص المدمجة قد أعدت دون أية تغيرات



يجب أن

تحفظ هذه الأقراص المدمجة بعيدا عن الغبار



MONA MAGHRABY



كلية العلوم - قسم الكيمياء



" Radiation Synthesis of Silica Aerogel Reinforcement by Polymers as Thermal Insulator Material "

A Thesis

"Submitted for the degree of Master of Science As partial
fulfillment for requirements of the master science"

"Amr Mohamed Osman Abd El-Malek"

First Dr.Nour El-din Ahmed AbdEl-Satar

Associated Professor of organic chemistry
Faculty of Science
Ain Shams University.

Second Dr. Amira.Abd El-Halim Elsayed

Associated Professor of organic chemistry
Faculty of Science
Ain Shams University.

Third Dr.Mohamed Mohamady Ghobashy

Associated Professor of organic chemistry
Radiation Research of Polymer chemistry
Atomic Energy Authority.

Department of chemistry

Faculty of science

Ain shams university

2021



كلية العلوم - قسم الكيمياء



Radiation Synthesis of Silica Aerogel Reinforcement by Polymers as Thermal Insulator Material

By

Amr Mohamed Osman Abd El-Malek

Thesis Advisors

Approved

First Dr.Nour El-din Ahmed AbdEl-Satar

Associated Professor of organic chemistry
Faculty of Science
Ain Shams University.

Second Dr. Amira.Abd El-Halim Elsayed

Associated Professor of organic chemistry
Faculty of Science
Ain Shams University

Third Dr.Mohamed Mohamady Ghobash

Associated Professor of chemistry
Radiation Research of Polymer chemistry
Atomic Energy Authority.

Head of Chemistry Department

Prof. Dr.Ayman Ayoub Abdel-Shafi

Acknowledgements

I'd like to show my gratitude to Dr. Nour El-din Ahmed, Associated professor of chemistry at Ain Shams University's Faculty of Science, for his courageous actions, constant guidance, support and invaluable assistance with practical work and thesis writing.

I'd like to thank Dr. Amira Abd El-Halim, Associated professor of chemistry at Ain Shams University's Faculty of Science, for her assistance and support.

Dr. Mohamed Mohamdy Ghobashy, professor of chemistry, Radiation Research of Polymer Chemistry, Atomic Energy Authority, who deserves our deepest gratitude, through his tireless efforts, constant direction, encouragement and invaluable assistance in practical work and thesis writing, he made this work possible. He was more than a boss; he was a constant source of encouragement and support for me. His wise counsel constantly pushed me onward.

I want to express my gratitude to my father and brothers for their constant support and prayers.

Finally, every successful man has a great woman by his side, and I have two: my wife and mother. I appreciate everything they've put up with in order to put up with and assist me in finishing my goal.

Amr Mohamed osman

CONTENTS

List of Contents

Subject	page
Aim of Work -----	I
List of Abbreviation -----	II
List of Tables -----	III
List of Figure -----	IV
I.Abstract -----	VI
II.Summary -----	VII
III.Introduction -----	1
IV.Results and Disssusion -----	26
- The microstructure of the Ca-Silicate and Al-Silicate aerogel -----	26
- The density and porous volume (%) of Ca-Silicate and Al-Silicate aerogel-----	39
- The Swelling property of Si-PAAm hydrogel samples-----	42
- FTIR Spectroscopy analysis -----	45
- Thermal conductivity performed for (Ca-Si) and (Al-Si) aerogel --	50
V.Expermintal -----	54
- Material -----	54
- Methods of preparation of silica PAAm hydrogel-----	54
- Measurments of swelling properties-----	59
- The density and pore volume (%) of Ca-silica and Al-silica aerogel at different PAAm contents -----	60
- Scanning electron microscope (SEM) -----	61
- Fourier-transform infrared (FTIR) spectroscopy -----	62
-Thermal conductivity measurement -----	63
VI.Conclusion -----	67
VII.Refernces -----	68
VIII.Arabic summary -----المخلص العربي	89
المستخلص -----	90

Aim of the Work

Aim of the Work

The present study was designed to prepare aerogels through cross-linked polyacrylamide (PAAm) hydrogel was reported through the use of gamma irradiation technique. Hydrogel obtained from gamma irradiation of acrylamide monomer dissolved in a solution of sodium silicate as a silicon precursor. Various irradiation doses (10 up to 60 kGy) and various acrylamide (AAm) contents (6.25, 9.37, 12.5, and 30%) were utilized in the polymerization process to investigate higher modulus as reinforced silica aerogels of low density. The reinforced Ca-silicate and Al-silicate aerogels can be an outstanding thermal insulating material used for different industrial and space exploration, with their very porous texture. the most easy techniques of mechanical reinforcing result in increased density and thus increased heat conductivity This is due to the requirement to increase the total number of connection sites inside the silica aerogel, which has resulted in an increase in the total amount of material utilised for the manufacturing of the gel matrix. Recent accomplishments include the creation of aerogels with a three-order-of-magnitude increase in maximum compression strength at break while only doubling the density and thermal conductivity. As a result, the goal of this paper is to provide an overview of several approaches for increasing the strength or stiffness of silica aerogel monoliths while reducing their density and thermal conductivity .

List of Abbreviation

NO	Symbol	Abbreviation
1	kGy	Kilo Gray
2	PAAm	Poly acrylamide
3	Ca-Si	Calcium – Silicate
4	Al-Si	Aluminium - Silicate
5	Si-PAAm	Silica polyacrylamide
6	TEM	Transmission electron microscop
7	KD₂	Thermal properties analyzer
8	EVA	Extra-vehicular activity
9	EDL	Entry,descent and landing
10	ATIS	Aerogel thin insulation sheets
11	TEOS	Tetraethyl orthosilicate
12	TMOS	Tetramethyl oxysilane
13	APD	Ambient pressure drying
14	TMCS	Trimethyl chloro silane
15	HMDZ	Hexamethyl disilazane
16	HLP	Hydrophile-lipophile balance
17	ICF	Internal confinement fusion
18	LSTS	Low surface tension solvent
19	MTMS	Methyl trimethoxy silane
20	AGU_s	Aerogel glazing units
21	SCD	Super critical drying
22	CNF	Cellulose nano fiber
23	SF	Silk fibroin
24	EDEX,EDS,SEM	Energy dispersive X-ray analysis
25	SE,BSE,TE	X–ray electromagnetic radition
26	SDD	Silicon-drift detectors
27	FE-SEM	Field emission-scan electron microscop

List of Tables

Table No.	Heading	Page No.
1	The physical properties of Ca-Si aerogels with various PAAm contents and irradiation doses.	41
2	The physical properties of Al-Si aerogels with various PAAm contents and irradiation doses.	42
3	Results of thermal conductivity of Ca-silica aerogel.	51
4	Results of thermal conductivity of Al-silica aerogel	53
5a	Keywords of the prepared Si-PAAm and Ca-Si aerogels samples with concentration 6.25% calcium silicate	55
5b	Keywords of the prepared Si-PAAm and Al-Si aerogels samples with concentration 6.25% aluminum silicate.	56
5c	Keywords of the prepared Si-PAAm and Ca-Si aerogels samples with concentration 9.37% calcium silicate.	56
5d	Keywords of the prepared Si-PAAm and Al-Si aerogels samples with concentration 9.37% aluminum silicate.	57
5e	Keywords of the prepared Si-PAAm and Ca-Si aerogels samples with concentration 12.5 % calcium silicate .	57
5f	Keywords of the prepared Si-PAAm and Al-Si aerogels samples with concentration 12.5% aluminum silicate.	58
5j	Keywords of the prepared Si-PAAm and Ca-Si aerogels samples with concentration 30% calcium silicate .	58
5k	Keywords of the prepared Si-PAAm and Al-Si aerogels samples with concentration 30% aluminum silicate .	59

List of Figures

Figure No.	Legend	Page No.
1	Preparation procedure of the calcium silicate aerogels.	viii
2	Preparation procedure of the Aluminium silicate aerogel	iiv
3	Nanometre-scale particles and pores in an aerogel. (a) Network architecture of an aerogel. (b) Electron micrograph of a silica aerogel.	6
4	Aerogel ‘puffed-up sand’ or ‘frozen smoke’	6
5	(a) Transmission electron microscopy (TEM) picture of silica aerogel from Tetraethyloorthosilane (TEOS) super critically dried in CO ₂ ; (b) adsorption/desorption isotherms of N ₂ for silica aerogel (curve shape corresponds to the characteristic mesoporous structure).	16
6	(a) Scanning electron microscopy (SEM) picture of silica aerogel blanket; (b) Silica aerogel granules.	16
7	The 1d diffusion preparation of gradient Fe(II)/SiO ₂ gels is depicted schematically.	23
8	A typical EDX spectrum shows the number of counts on the y-axis and the energy of the X-rays on the x-axis. The height of the peaks aids in the quantification of each element's concentration in the sample and the position of the peaks aids in the identification of the elements.	27
9	showEDX is used to generate X-rays in a two-step method. The energy delivered to the atomic electron first knocks it off, leaving a hole behind. Second, another electron from a higher energy shell fills its place, causing the characteristic X-ray to be produced	28
10	The SEM images of Ca-Si aerogels of (a) 6Ca-Si10, (b) 6Ca-Si30, (c) 6Ca-Si60, (d) 9Ca-Si10, (e) 9Ca-Si30, (f) 9Ca-Si60, (g) 12Ca-Si10, (h) 12Ca-Si30, (k) 12Ca-Si60, (l) 30Ca-Si10, (m) 30Ca-Si30, and (n) 30Ca-Si60 with different PAAm solid contents and irradiation doses.	31-32

11	The EDX charts of Ca-(Si-PAAm) ignited at different temperatures of (a) 25 °C, (b) 250 °C, and (c) 1100 °C.	34
12	The SEM images of Al-Si aerogels of (a) 6Al-Si10, (b) 6Al-Si30, (c) 6Al-Si60, (d) 9Al-Si10, (e) 9Al-Si30, (f) 9Al-Si60, (g) 12Al-Si10, (h) 12Al-Si30, (k) 12Al-Si60, (l) 30Al-Si10, (m) 30Al-Si30, and (n) AlCa-Si60 with different PAAm solid contents and irradiation doses.	36-37
13	The EDX charts of Al-(Si-PAAm) ignited at different temperatures of (a) 25 °C, (b) 250 °C and (c) 1100 °C.	39
14	show increasing of density with decrease of solid content	41
15	show increasing of density with decrease of solid content	42
16	The swelling degree at 25 °C of Si-PAAm hydrogels discs with different PAAm contents of (a) 6.25, (b) 9.37, (c) 12.5 and (d) 30 (%).	44
17	The maximum swelling (g/g) of Si-PAAm hydrogels with different PAAm contents of 6.25, 9.37, 12.5, and 30 (%)vs. irradiation doses.	45
18	The FTIR of Ca-(Si-PAAm) ₂₅ Ca-(Si-PAAm) ₂₅₀ , and Ca-(Si-PAAm) ₁₁₀₀ at temperatures of (a) 25,(b) 250, and (c)1100 °C respectively.	46-47
19	The FTIR of Al-(Si-PAAm) ₂₅ , Al-(Si-PAAm) ₂₅₀ , and Al-(Si-PAAm) ₁₁₀₀ at temperatures of (a) 25,(b) 250, and (c)1100 °C respectively.	49-50
20	Images of 30(ca-Si) aerogel (a) before and (b) during ignition byBunsen burner	51
21	Images of 30(Al-Si) aerogel (a) before and(b) during ignition byBunsen burner	52
22	Thermal conductivity analyzer KD ₂ pro	63
23	The thermal conductivity of Ca-Si and Al-Si aerogels was measured using the KD ₂ Pro thermal conductivity equipment, which included a transient line heat source .	66