MARMARA UNIVERSITY
INSTITUTE FOR GRADUATE STUDIES
IN PURE AND APPLIED SCIENCES

PROCESSING AND CHARACTERIZATION OF FOAM GLASS

AYLİN ŞAHİN

MASTER THESIS

Department of Metallurgical and Materials Engineering

Thesis Supervisor
Prof. Dr. Recep ARTIR

ISTANBUL, 2017

MARMARA UNIVERSITY
INSTITUTE FOR GRADUATE STUDIES
IN PURE AND APPLIED SCIENCES

PROCESSING AND CHARACTERIZATION OF FOAM GLASS

AYLİN ŞAHİN

(524714001)

MASTER THESIS

Department of Metallurgical and Materials Engineering

Thesis Supervisor

Prof. Dr. Recep ARTIR

ISTANBUL, 2017

ACKNOWLEDGEMENT

I would firstly like to express my endless thanks to Prof. Dr. Recep ARTIR, my supervisor, for all his extensive helps, academic advices and guidance throughout the duration of my M.Sc. I am very grateful for his precious efforts and instructions at every stage of my thesis. I would also like to express my massive thanks to Dr. Mustafa KARA and Dr. Yasemin KILIÇ for their contributions and endless helps from beginning to the end of my study. Their unsparing and precious supports were of great value for me.

In addition, I would like to present my grateful thanks to my precious friend Ekin KAHRAMAN who is always behind me, and also İrem YÜCEL, Sena DALOĞLU, Aybüke POLATKAN and Merve TÜREMİŞ for their limitless encouraging. Also, many thanks to my colleagues Hidayet BODUR and Erbay KELEŞ who always heart and soul in help and support my laboratory studies. I would also thank to TÜBİTAK Marmara Research Centre Materials Institute which supplied laboratory infrastructure and made me possible to carry out my laboratory studies and tests, also many thanks to those who contributed my other colleagues who conducted and help making my analyses. Moreover, I would like to express my deepest thanks to Prof. Dr. Lütfullah GÜNDÜZ from İzmir Katip Çelebi University, Prof. Dr. Alpagut KARA, Zehra Emel OYTAÇ and Mustafa ÇOBANCI from Eskişehir Ceramic Research Centre for their kindly helps and making some analysis in their institutes without any materialistic expectation.

Finally, I would like to present my special thanks to my best friend and sister Gülçin ŞAHİN and my lovely parents who encourage and always stand behind me in every stage of this period and my life. I am grateful for their endless supports and sensibilities, without their support and encourage, this thesis would not have been completed.

November, 2017 **Aylin ŞAHİN**

TABLE OF CONTENTS

ACKNOWLEDGEMENT ..... **i**
TABLE OF CONTENTS ..... **ii**
ÖZET ..... **vi**
ABSTRACT ..... **vii**
SYMBOLS ..... **viii**
ABBREVIATIONS ..... **xii**
LIST OF FIGURES ..... **xiii**
LIST OF TABLES ..... **xix**
CHAPTER 1 – INTRODUCTION AND AIM **1**
1.1. General Background 3
1.1.1. Thermal insulation 4
1.1.2. Thermal transmittance (U-value) 5
1.1.3. Thermal insulation materials 7
1.2. Foam Glass 11
1.2.1. History of foam glass 11
1.2.2. Definition of foam glass 11
1.2.3. Properties of foam glass 12
1.2.4. Application area of foam glass 15
1.3. Foam Glass Production Process 16
1.3.1. Continuous sheet process for loose foam glass agregates 16
1.3.2. Continuous process for foam glass blocks and different shapes 17
1.3.3. Spherical pellet production 18
1.4. Important Parameters in Foam Glass Production 18
1.4.1. Glass 19
1.4.2. Foaming agents 20
1.4.2.1. Reducing agents 21
1.4.2.2. Decomposable agents 22
1.4.3. Foaming mechanism 22
1.4.3.1. Foaming by reaction 23
1.4.3.2. Foaming by thermal decomposition 24

1.4.4. Heating rate 24
1.4.5. Foaming temperature 25
1.4.6. Heating time 26
1.4.7. Cooling rate 26
1.4.8. Initial particle size of raw materials 26
1.5. Literature Survey 28
CHAPTER 2 - MATERIAL AND METHOD 37
2.1. Raw Materials 37
2.1.1. Glass powder 37
2.1.2. SiC powders with different purity grades 39
2.1.3. Binder 40
2.2. Characterization Methods of Raw Material 40
2.2.1. Particle size distribution 40
2.2.2. Differential thermal analysis (DTA) - thermal gravimetric analysis (TGA) 41
2.2.3. X-Ray Fluorescence (XRF) – semi quantitative elemental analysis 42
2.2.4. Mineralogical analysis (XRD) 42
2.2.5. Scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) 43
2.3. Experimental Procedure 43
2.3.1. Flow chart 44
2.3.2. Sample preparation 44
2.3.3. Granulation and pressing procedure 45
2.3.4. Sintering process 46
2.4. Characterization Methods of Foam Glass Samples 47
2.4.1. Volume expansion ratio 48
2.4.2. Mineralogical analysis (XRD) 49
2.4.3. Compressive strength 49
2.4.4. Apparent density 50
2.4.5. Short term water absorption by partial immersion 51
2.4.6. Long term water absorption by total immersion 52
2.4.7. Microstructural evaluation (SEM) 53

2.4.8. Thermal conductivity 53
2.4.9. Total porosity 54
2.4.10. Coefficient of thermal expansion 55
2.4.11. Water vapor diffusivity strength 56
CHAPTER 3 - RESULTS & DISCUSSION 57
3.1. Characterization Results and Discussion of Raw Materials 57
3.1.1. Particle size distribution analysis 57
3.1.2. Differential thermal analysis (DTA) - Thermal gravimetric analysis (TGA) 59
3.1.3. X-Ray Fluorescence (XRF) - semiquantitative elemental analysis 62
3.1.4. Mineralogical analysis (XRD) results of raw materials 64
3.1.5. Scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) investigation of SiC raw materials 66
3.2. Foam Glass Samples 69
3.2.1. 90 % Purity graded SiC foam glass samples 69
3.2.2. 95 % Purity graded SiC foam glass samples 72
3.2.3. 98 % Purity graded SiC foam glass samples 75
3.3. Characterization Results and Discussion of Foam Glass Samples 78
3.3.1. Volume expansion ratios 78
3.3.2. Mineralogical analysis (XRD) 81
3.3.3. Compressive strength 84
3.3.4. Apparent density 89
3.3.5. Short term water absorption by partial immersion 93
3.3.6. Long term water absorption by total immersion 97
3.3.7. Microstructural evaluation (SEM) 100
3.3.8. Thermal conductivity 104
3.3.9. Total porosity 108
3.3.10. Coefficient of thermal expansion 113
3.3.11. Water vapor diffusion resistance 116
3.4. General Results and Discussion 119

CHAPTER 4 - CONCLUSION 124
FUTURE WORK..... 126
REFERENCES 127
CURRICULUM VITAE 135

ÖZET

CAM KÖPÜK ÜRETİMİ VE KARAKTERİZASYONU

Bu çalışmanın amacı, farklı saflık dereceleri (%), kompozisyonlar (ağ. %) ve sinterleme sıcaklıkları (°C) kullanılarak cam köpük malzemesinin geliştirilmesine yönelik üretim yöntemlerinin araştırılmasıdır. Birincil hammadde olarak düz cam atığı tozları kullanılmış olup cam köpük üretiminde temel teşkil eden ve köpüklenme reaksiyonlarını sağlayan köpükleyici ajan olarak farklı saflıklarda SiC tozları kullanılmıştır. Bu çalışmada üç parametrenin etkisi araştırılmıştır: köpükleyici ajan saflık derecesi (% 90, 95 ve 98), köpükleyici ajan katkı oranı (ağ. % 1, 3, 5, 7 ve 9) ve sinterleme sıcaklığı (800, 825, 850 ve 875°C). Cam ve SiC tozları farklı kompozisyonlara göre karıştırılmış, daha sonra preslenerek her bir sıcaklıkta sinterlenmiştir. Cam köpük numuneleri bir parametre değiştirilirken diğer parametreler sabit tutularak üretilmiştir. Elde edilen numuneler ilgili standartlarda istenilen özelliklere bağlı olarak karakterize edilmiştir. Bu kapsamda, hacimsel genleşme oranı, mineralojik analiz, görünür yoğunluk, basma mukavemeti, kısa süreli su emme, uzun süreli su emme, SEM incelemesi, termal iletkenlik, toplam porozite, termal genleşme katsayısı ve su buharı difüzyon direnci testleri yapılmıştır. Araştırılan bu üç parametrenin cam köpüklerin karakteristik özellikleri üzerinde oldukça etkili olduğu görülmüştür. Elde edilen cam köpük numuneleri ilgili standartlarda belirtilen ve istenilen özellikleri karşılamıştır.

Sonuç olarak, yoğunluk ve termal iletkenliği düşük, porozitesi yüksek olmasının yanında yeterli basma dayanımına sahip cam köpük numuneleri üretilmiştir. Bu çalışmanın özgün tarafı, literatür çalışmalarından farklı olarak % 90 saflıkta SiC kullanarak ve 850°C gibi dikkate değer düşük bir sıcaklıkta sinterlenerek üretilen cam köpük numunelerinde istenilen düşük yoğunluk ve düşük termal iletkenlik değerlerinin elde edilebilmiş olmasıdır. Bu çalışma, atıktan değerli bir izolasyon malzemesine dönüştürülebilme potansiyeli açısından cam atıkları için uygun bir kullanım alanı da sunmaktadır. Sonuç olarak, bu çalışma ithal olarak elde edilen ve insan sağlığına uygun olmayan polimer bazlı izolasyon malzemeleri yerine çeşitli yalıtım sistemlerinde kullanılabilecek ve istenilen özellikleri sağlayan cam köpük malzemesinin üretilebileceğini göstermiştir.

ABSTRACT

PROCESSING AND CHARACTERIZATION OF FOAM GLASS

The aim of this study is that developing a foam glass material by investigating production methods with using different purity grades (%), compositions (wt. %) and sintering temperatures (°C). Sheet glass waste powders were primary raw material, while SiC powders with different purity graded were used as foaming agent which is the essential to produce foam glass with supplying foaming reactions. Effects of the three parameters were investigated in this study: foaming agent purity grade (90, 95 and 98 %), foaming agent addition ratio (1, 3, 5, 7 and 9 wt. %) and sintering temperature (800, 825, 850 and 875°C). Glass and SiC powders were mixed according to different compositions, then pressed and sintered at each temperature. Foam glass samples were achieved with changing one parameter during other parameters was constant. Obtained samples were characterized based on the required properties according to related standard. Within this scope, volume expansion ratio, mineralogical analysis, apparent density, compressive strength, short term water absorption, long term water absorption, SEM investigation, thermal conductivity, total porosity, thermal expansion coefficient and water vapor diffusion resistance tests were applied. It was revealed that researched three parameters are highly effective on foam glass characteristics. Obtained foam glass samples met the required properties which were declared in the related standards.

Finally, various foam glass products were achieved with having low density, low thermal conductivity and high porosity beside their sufficient compressive strength. It was challenged as distinct results from the literature by using 90 % purity graded SiC and sintering at 850°C which is considerably lower temperature for achieving such a low density and low thermal conductivity properties. The study also suggests that a suitable usage area for waste glass because of the capability of its transforming from a waste into a valuable insulation material. Moreover, an alternative insulation material is presented for using at the building insulation which is essential for energy efficiency in the recent years. In conclusion, this study proposes that foam glass materials can be produced with desired properties and can be used at various insulation systems rather than importing polymer based unhealthy insulation materials.

SYMBOLS

Al : Aluminum

Al_2O_3 : Aluminum oxide (alumina)

A_p : Bottom surface area of the test specimen for water absorption test

As_2O_3 : Arsenic trioxide

BaO : Barium oxide

B_2O_3 : Boron trioxide

C : Carbon

Ca : Calcium

CaF_2 : Calcium difluoride

CaO : Calcium oxide

$CaCO_3$: Calcium carbonate

$CaSiO_3$: Calcium metasilicate

Cl : Chlorine

CO : Carbon monoxide

CO_2 : Carbon dioxide

Cr : Chromium

CuO : Cupric oxide (Copper(II) oxide)

D : Diameter of pore

d : Thickness

dT/dx : The amount of temperature change in x direction

F : Fluorine

Fe : Iron

Fe_2O_3 : Ferric oxide (Iron(III) oxide)

g/cm^3 : Gram per cubic centimetre

g/m^2h : Water vapor flow intensity

h : Hour

Hg : Mercury

K : Potassium

k : Heat transfer coefficient (W/mK)

kg : Kilogram

kg/m^2 **:** Kilogram per square meter

$kg/(m^2.sec.Pa)$: Water vapor transmission

K^{-1} **:** Coefficient of thermal expansion

K_2O **:** Potassium oxide

Mg **:** Magnesium

$MgCO_3$ **:** Magnesium carbonate

MgO **:** Magnesium oxide (magnesia)

Mn **:** Manganese

MnO **:** Manganese oxide

MnO_2 **:** Manganese dioxide

MPa **:** Mega pascal

N_2 **:** Nitrogen

Na **:** Sodium

Ni **:** Nickel

O **:** Oxygen

m **:** Mass of the test specimen

m_0 **:** Initial mass of the test specimen

m_{24} **:** Mass of the test sample after partial immersion for 24 h

m_{28} **:** Mass of the test specimen after total immersion for 28 days

m **:** Meter

min : Minute

mm **:** Milimeter

mL **:** Milliliter

Na_2CO_3 **:** Sodium carbonate

Na_2O **:** Sodium oxide

$NH_4H_2PO_4$ **:** Ammonium dihydrogen phosphate

NiO **:** Nickel(II) oxide

P **:** Phosphorus

P **:** Applied pressure

Pa.s **:** Pascal-second

Pb **:** Lead

PbO **:** Lead monoxide (Lead(II) oxide)

P_2O_5 **:** Phosphorus pentoxide

psi **:** Pounds per square

Q **:** Amount of heat passing in the x direction

R **:** Resistance of thermal insulation (m^2K/W)

S **:** Sulphur

s **:** second

Sb_2O_3 **:** Antimony trioxide

Si **:** Silicon

SiC **:** Silicon carbide

Si_3N_4 **:** Silicon nitride

SiO_2 **:** Silicon dioxide (silica)

SO_3 **:** Sulfur trioxide

Sr **:** Strontium

SrO **:** Strontium oxide

T **:** Ambient temperature

Ti **:** Titanium

TiO_2 **:** Titanium dioxide

TiN **:** Titanium nitride

U **:** Thermal transmittance (W/m^2K)

V **:** Volume of the test specimen

V **:** Vanadium

V_2O_5 **:** Vanadium pentoxide (Vanadium(V) oxide)

W **:** Watt

W_p **:** Short term partial immersion for water absorption

W_{lt} **:** Long term total immersion for water absorption

Zn **:** Zinc

ZnO **:** Zinc oxide

Zr **:** Zirconium

ZrO_2 **:** Zirconium dioxide (zirconia)

Å **:** Angstrom

λ **:** Coefficient of the thermal conductivity

μ **:** Coefficient of water vapor diffusion resistance

μm : Micrometer

ϕ : Relative density

Ø : Diameter

Q : Ambient relative humidity (%)

ρ_a : Apparent density (g/cm^3)

ρ_w : Density of water (1000 kg/m^3)

γ : Surface tension of mercury

θ : Contact angle between mercury and pore wall

δ : Water vapor permeability (kg/(m.sec.Pa))

δ_a : Water vapor permeability of air (kg/(m.sec.Pa))

ABBREVIATIONS

CEN **:** Comité Européen de Normalisation (European Committee for Standardization)

CMC **:** Carboxymethyl cellulose

CRT **:** Cathode ray tube

DS **:** Dimensional stability

DTA **:** Differential thermal analysis

ECB **:** Expanded cork boards

EPB **:** Expanded perlite boards

EPS **:** Expanded polystyrene

JCPDS **:** Joint Committee on Powder Diffraction Standarts

ISO **:** International Organization for Standardization

N/A **:** Not applicable

PF **:** Phenol foam

psi **:** Pounds per square inch

PUR **:** Polyurethane foam

PVC **:** Polyvinyl chloride

rpm **:** Revolutions per minute (rev/min)

SEM **:** Scanning electron microscope

SI **:** The international system of units

USD **:** United States dollar

TGA **:** Thermal gravimetric analysis

XPS **:** Extruded polystyrene

XRD **:** X-Ray diffractometer

XRF **:** X-Ray fluorescence

LIST OF FIGURES

Figure 1.1. Energy consumption in Turkey. 3
Figure 1.2. The ratio of energy consumption in buildings. 4
Figure 1.3. Heat losses at multi and single storey buildings. 5
Figure 1.4. Design and places of construction elements. 7
Figure 1.5. Illustration of thermal insulation materials used in Turkey; (a) extruded polystyrene (XPS), (b) expanded polystyrene (EPS), (c) Fiber glass, (d) stone wool, (e) polyurethane foam (PUR), (f) phenol foam (PF), (g) expanded perlite boards (EPB), (h) expanded cork boards (ECB) and (i) foam glass 10
Figure 1.6. Illustration of some foam glass products. 12
Figure 1.7. Illustration of required properties of foam glass. 14
Figure 1.8. Some examples for foam glass applications. 15
Figure 1.9. (a) Loose aggregates, (b) foam glass blocks and (c) foam glass pellets. 16
Figure 1.10. (a) Foam glass sheet furnace, (b) production of loose aggregate. 17
Figure 1.11. (a) Molten glass and (b) foam glass block production within mold. 18
Figure 1.12. Foam glass pellets (a) production by rotary furnace and (b) products. ...18
Figure 1.13. Microstructures of foam glass samples produced from funnel glass with addition of (a) 1 wt. % SiC and (b) 5 wt. % SiC at same sintering schedule 22
Figure 1.14. Foam glass samples produced at (a) 750°C, (b) 850°C and (c) 950°C 25
Figure 1.15. Apparent density of foam glass as a function of thermal treatment time. 26
Figure 1.16. Effect of glass powder grain size on (a) microstructure, (b) pore diameter of foam glass 27
Figure 1.17. Foam glass samples produced by Tulyaganov et al. 29
Figure 1.18. SEM micrograph of foam glass produced with (a) 5 wt. % SiC and (b) 5 wt. % TiN. 33
Figure 2.1. Waste sheet glass processing steps (a) waste sheet glass, (b) crushing step, (c) crushed glass cullet, (d) jaw crusher, (e) coarse crushed

glass, (f) ball mills for wet milling, (g) dried glass after milling and (h) fine glass powder. 38
Figure 2.2. 90 % purity graded SiC powder. 39
Figure 2.3. 95 % purity graded SiC powder. 39
Figure 2.4. 98 % purity graded SiC powder. 39
Figure 2.5. Diluted carboxymethyl cellulose binder. 40
Figure 2.6. Malvern Mastersizer-2000 device 41
Figure 2.7. SEIKO ExStar 6300 model DTA/TG thermal analysis device. 41
Figure 2.8. SHIMADZU XRD-6000 Diffractometer device. 43
Figure 2.9. Flow chart of experimental study. 44
Figure 2.10. (a) Plastic containers, (b) alumina balls and (c) ball milling device. 45
Figure 2.11. Granulation process (a) addition of binder, (b) granulation, (c) sieving and (d) granulated sample. 45
Figure 2.12. Hydraulic press. 46
Figure 2.13. Some of the pressed samples. 46
Figure 2.14. PROTHERM PLF 120/45 model furnace (a) outside view, (b) inside view before sintering and (c) inside view after sintering. 47
Figure 2.15. Measuring of foam glass sample volume (a) before and (b) after sintering 48
Figure 2.16. Some foam glass samples prepared for mechanical tests (a) 98 % purity graded SiC with a ratio of 5 wt. %, (b) 95 % purity graded SiC with a ratio of 7 wt. % sintering at 800, 825, 850 and 875°C, respectively. 49
Figure 2.17. (a) Zwick Z250SN5A device and (b) an example during compressive test. 50
Figure 2.18. Illustration of short term partial immersion test. 51
Figure 2.19. Illustration of drainage process. 52
Figure 2.20. Illustration of thermal conductivity within a cylindrical material. 54
Figure 2.21. NETZSCH DIL 402 C/3/F model dilatometer device. 56
Figure 3.1. Particle size distribution of waste sheet glass powders. 58
Figure 3.2. Particle size distribution of 90 % purity graded SiC powders. 58
Figure 3.3. Particle size distribution of 95 % purity graded SiC powders. 59

Figure 3.4. Particle size distribution of 98 % purity graded SiC powders. 59
Figure 3.5. DTA/TG Analysis result of waste sheet glass. 60
Figure 3.6. DTA/TG Analysis result of 90 % purity grade SiC. 61
Figure 3.7. DTA/TG Analysis result of 95 % purity grade SiC. 61
Figure 3.8. DTA/TG Analysis result of 98 % purity grade SiC. 62
Figure 3.9. XRD pattern of waste sheet glass powders. 64
Figure 3.10. XRD pattern of 90 % purity graded SiC powders. 65
Figure 3.11. XRD pattern of 95 % purity graded SiC powders. 65
Figure 3.12. XRD pattern of 98 % purity graded SiC powders. 66
Figure 3.13. EDS analysis results of (a) 90 % purity graded SiC powder; (b) 95 % purity graded SiC powder and (c) 98 % purity graded SiC powder. 68
Figure 3.14. Illustration of foam glass samples with 90 % purity graded SiC addition before sintering and after sintering at 800, 825, 850 and 875°C with a ratio of (a) 1 wt.%; (b) 3 wt.%; (c) 5 wt.%; (d) 7 wt.%; (e) 9 wt.%, respectively. 70
Figure 3.15. Illustration of crosscut foam glass samples with 90% purity graded SiC after sintering at (a) 800°C; (b) 825°C; (c) 850°C ; (d) 875°C, with a ratio of 1, 3, 5, 7 and 9 wt.%, respectively.....71
Figure 3.16. Illustration of foam glass samples with 95 % purity graded SiC addition before sintering and after sintering at 800, 825, 850, 875°C with (a) 1 wt.%; (b) 3 wt.%; (c) 5 wt.%; (d) 7 wt.%; (e) 9 wt.%, respectively. 73
Figure 3.17. Illustration of crosscut foam glass samples with 95% purity graded SiC after sintering at (a) 800°C; (b) 825°C; (c) 850°C ; (d) 875°C, with a ratio of 1, 3, 5, 7 and 9 wt.%, respectively.....74
Figure 3.18. Illustration of foam glass samples with 98 % purity graded SiC addition before sintering and after sintering at 800, 825, 850 and 875°C with a ratio of (a) 1 wt.%; (b) 3 wt.%; (c) 5 wt.%; (d) 7 wt.%; (e) 9 wt.%, respectively. 76
Figure 3.19. Illustration of crosscut foam glass samples with 98 % purity graded SiC after sintering at (a) 800°C; (b) 825°C; (c) 850°C ; (d) 875°C, with a ratio of 1, 3, 5, 7 and 9 wt.%, respectively.....77

Figure 3.20. Volume expansion graph of foam glass samples with 90 % purity graded SiC with different addition ratios and different sintering temperatures. 79
Figure 3.21. Volume expansion graph of foam glass samples with 95 % purity graded SiC with different addition ratios and different sintering temperatures. 79
Figure 3.22. Volume expansion graph of foam glass samples with 98 % purity graded SiC with different addition ratios and different sintering temperatures. 80
Figure 3.23. Mineralogical phase patterns of foam glass with 1, 5 and 9 wt. % SiC (90 % purity) and sintered at 850°C (90-1-C, 90-5-C and 90-9-C, respectively). 82
Figure 3.24. Mineralogical phase patterns of foam glass with 5 wt. % SiC (90 % purity) and sintered at 800, 825, 850 and 875°C (90-5-A, 90-5-B, 90-5-C and 90-5-D, respectively). 83
Figure 3.25. Mineralogical phase patterns of foam glass with 5 wt. % SiC (90, 95 and 98 % purity) and sintered at 850°C (90-5-C, 95-5-C and 98-5-C, respectively). 83
Figure 3.26. Compressive strength graph of foam glass samples with 90 % purity graded SiC with various addition ratios and sintering temperatures. 85
Figure 3.27. Compressive strength graph of foam glass samples with 95 % purity graded SiC with various addition ratios and sintering temperatures. 85
Figure 3.28. Compressive strength graph of foam glass samples with 98 % purity graded SiC with various addition ratios and sintering temperatures. 86
Figure 3.29. Comparison of foaming agent addition effects on cell size for fired foam glass samples with a ratio of (a) 1 wt. % and (b) 9 wt. %. 86
Figure 3.30. Comparison of sintering temperature effects on cell size for fired foam glass samples having same composition which sintered at (a) 800°C and (b) 875°C. 87
Figure 3.31. Comparison of foaming agent purity effects on cell size for fired foam glass samples with (a) 90 % purity graded SiC and (b) 98 % purity graded SiC. 88

Figure 3.32. Apparent density variation of 90% grade SiC addition ratio of foam glass samples.....90
Figure 3.33. Apparent density variation of 95% grade SiC addition ratio of foam glass samples.....91
Figure 3.34. Apparent density variation of 98% grade SiC addition ratio of foam glass samples.....92
Figure 3.35. Short term water absorption by partial immersion graph of the selected foam glass samples sintered at 800°C.....94
Figure 3.36. Short term water absorption graph of the selected foam glass samples sintered at 850°C.....95
Figure 3.37. Short term water absorption by partial immersion graph of the foam glass samples with using 90 % purity graded SiC with a ratio of 5 wt. % at 800, 825, 850 and 875°C. 96
Figure 3.38. Long term water absorption by total immersion (%) graph of the selected foam glass samples sintered at 800°C.....97
Figure 3.39. Long term water absorption by total immersion graph of the selected foam glass samples sintered at 850°C.....98
Figure 3.40. Long term water absorption by total immersion graph of the foam glass samples with 90 % purity graded SiC with a ratio of 5 wt. % at 800, 825, 850 and 875°C. 99
Figure 3.41. Porous structures of the selected foam glass samples observed by SEM. 101
Figure 3.42. Strut (cell wall) views and thickness of selected foam glass samples. ..103
Figure 3.43. Variation of thermal conductivity with sintering temperature for foam glass samples containing 5 wt. % SiC foaming agent (90 % purity graded).....105
Figure 3.44. Variation of thermal conductivity with foaming agent addition ratio for foam glass samples containing 1, 5 and 9 wt. % SiC foaming agent (90 % purity graded) at 850°C. 106
Figure 3.45. Variation of thermal conductivity with foaming agent purity for foam glass samples containing 90, 95 and 98 % SiC (5 wt. %) sintered at 850°C.....107

Figure 3.46. Pore sizes and structures of the samples with using (a) 1 wt. %, (b) 5 wt. % and (c) 9 wt. % SiC (90 % purity graded) and sintered at 850°C. 109
Figure 3.47. Total porosity variations of foam glass samples with 1, 5 and 9 wt. % SiC (90 % purity graded) foaming agent and sintered at 850°C.....109
Figure 3.48. Total porosity graph of foam glass samples produced with using 5 wt. % SiC (90 % purity graded) foaming agent and sintered at 850 and 875°C. 110
Figure 3.49. Pore structures of the samples with (a) 90 % purity, (b) 95 % purity and (c) 98 % purity graded SiC foaming agent with a ratio of 5 wt. % and sintered at 850°C. 111
Figure 3.50. Total porosity variations of foam glass samples with 90, 95 and 98 % purity graded SiC foaming agent (5 wt. %) and sintered at 850°C. 111
Figure 3.51. Top surface and bottom surface cell size distribution of foam glass samples with (a) 90 % and (b) 98 % purity graded SiC with a ratio of 3 wt. % and sintered at 850°C.....113
Figure 3.52. Coefficient of thermal expansion (K^{-1}) graph of the foam glass sample with 90 % purity graded SiC (5 wt. %) at 850°C. 114
Figure 3.53. Coefficient of thermal expansion (K^{-1}) graph of the foam glass sample with 95 % purity graded SiC (5 wt. %) at 850°C. 114
Figure 3.54. Coefficient of thermal expansion (K^{-1}) graph of the foam glass sample with 98 % purity graded SiC (5 wt. %) at 850°C. 115
Figure 3.55. The foam glass samples (90-5-C) used for water vapor resistance analysis.....117
Figure 3.56. Volume expansion - apparent density- compressive strength correlation graph of foam glasses. 122
Figure 3.57. Volume expansion - apparent density- total porosity correlation graph of foam glasses.....123
Figure 3.58. Total porosity- apparent density - thermal conductivity correlation graph of foam glasses.....123

LIST OF TABLES

Table 1.1. Calculated values of resistances of the internal and external faces. 6

Table 1.2. Comparison of foam glass and other thermal insulation materials used in Turkish market.....9

Table 1.3. Physical properties of foam glass. 13

Table 1.4. Comparison of the chemical compositions of some waste glasses from the literature. 20

Table 1.5. Possible reactions between SiC and gas species in various atmospheres.....24

Table 2.1. Raw materials used in the study.....37

Table 2.2. Parameters and different variance researched in the experimental study. 43

Table 2.3. Composition ratios of foam glass samples.....44

Table 2.4. Sintering schedules. 47

Table 3.1. Particle size results of glass and SiC raw materials. 58

Table 3.2. Semiquantitative elemental analysis results of waste sheet glass and SiC powders. 63

Table 3.3. Oxide contents of waste sheet glass powders. 63

Table 3.4. Mineralogical analysis (XRD) results of waste sheet glass and SiC powders. 64

Table 3.5. Microstructure images of SiC raw materials by using scanning electron microscope (SEM). 67

Table 3.6. 90 % Purity graded SiC foam glass sample codes. 69

Table 3.7. 95 % Purity graded SiC foam glass sample codes. 72

Table 3.8. 98 % Purity graded SiC foam glass sample codes. 75

Table 3.9. Volume expansion ratios (%) of foam glass samples after firing. 78

Table 3.10. Mineralogical phases detected after firing of foam glass samples. 82

Table 3.11. Compressive strength (MPa) values of foam glass samples. 84

Table 3.12. Apparent density (g/cm^3) results of foam glass samples. 89

Table 3.13. Short term water absorption values (kg/m^2) of selected samples. 94

Table 3.14. Short term water absorption values (kg/m^2) according to sintering temperatures. 96
Table 3.15. Long term water absorption values (%) of selected foam glass samples. 97
Table 3.16. Long term water absorption values (%) according to sintering temperature. 98
Table 3.17. Thermal conductivity (W/mK) values of selected foam glass samples. 104
Table 3.18. Total porosity values (%) of selected foam glass samples. 108
Table 3.19. Coefficient of thermal expansion (K^{-1}) values of selected foam glass samples. 114
Table 3.20. Water vapor diffusion resistance (μ) values of some materials. 116
Table 3.21. The results of the water vapor diffusion resistance (μ) test. 118
Table 3.22. Comparison of physical properties of selected foam glass samples with standard value and commercial product. 121

CHAPTER 1 – INTRODUCTION AND AIM

In recent years, energy consumption has increased depending on population growth in the world, and consequently available energy resources have rapidly depleted and due to fossil fuels consumption negative effect of CO_2 gas on the environment has significantly increased. It is also important that most part of energy being used in our country is imported. Energy efficiency is a concept to prevent the energy loss on gas, vapor, heat, air and electricity, also recycling and utilizing of various wastes or reducing energy requiring without production decline along by using advanced technology.

In Turkey, 30 % of the energy is consumed at the buildings while 85 % of this energy is used for heating purposes. However, nearly 50-60 % of the energy used in the buildings can be saved by applying thermal insulation. For this reason, one of the preventions which must be primarily applied on energy efficiency is making heat insulation of buildings [1].

Thermal insulation has become unavoidable in recent years because of the global climate change, rapid decreasing of energy resources on the earth and increasing the necessity of the controlling energy consumption for countries. Thermal insulation on buildings provides an improvement on energy saving, economy, safety and health issues with a clear reduction on environmental problems.

The most important feature of thermal insulation materials is coefficient of the thermal conductivity (λ). To classify a material as an insulation material, the coefficient of thermal conductivity (λ) value must be lower than 0.065 W/mK according to ISO and CEN standards [2]. Another significant property of thermal insulation materials is thermal transmittance, U value (W/m^2K) which depends on the coefficient of thermal conductivity (λ) and thickness in the direction of heat transfer. Calculation of U value based on the thickness of the construction material (d), coefficient of thermal conductivity (λ) and resistance of thermal insulation (R), (m^2K/W) [3]. It is indicated that smaller U value should be achieved to decrease the heat loss [4]. Thus, during selection of a thermal insulation material, consideration of λ and U values is quite important to achieve a satisfactory insulation.

Thermal insulation materials are mostly used at wall, roof, tiling, plumbing system and

window units. To choose the most proper thermal insulation material type, it should be well known the properties of the thermal insulation materials according to which area is going to be thermally insulated. In our country, fiber glass, stone wool, expanded polystyrene (EPS), extruded polystyrene (XPS), polyurethane foam (PUR), phenol foam (PF) and polyethylene foam are produced and used as thermal insulation material. However, *foam glass*, ceramic wool, polyisocyanate foam, polyvinyl chloride (PVC) foam, vermiculite and elastomeric rubber foam are imported as insulating material from abroad.

Foam glass is a remarkable material which has unique properties like lightweight, low density, low thermal conductivity, rigid structure with moderate compressive strength, non-flammability, freeze-resistance, chemically inert and nontoxic, rodent and insect-resistant, bacteria-resistant, and water and steam resistant [5]. In recent years, foam glass material has been preferred as an insulation material for buildings abroad because of its much better thermal insulation properties compared to the other traditional insulation materials. In addition, foam glass gives reliance on health and safety due to the main raw material is glass. Equivalent polymer based insulation materials are composed of mostly cancerogenic and easily flammable materials. Although these significant properties of foam glass, this material has not been produced in Turkey, yet. Some companies only import foam glass, however this material has not been used commonly in Turkey.

Throughout this study, detailed literature and market survey were conducted based on the foam glass production. From the literature and market survey, it has been realized that no company has attempted to produce foam glass in Turkey. The aim of this study is to investigate of production methods of foam glass with low thermal conductivity by using waste glass as main raw material confirming all requirements stated in the related standards that can be placed in the market. Within this scope, foam glass production methods and processing conditions were searched in the related literature such as firing temperatures; batch composition ratios and purity grades of foaming agents.

1.1. General Background

Throughout history, mankind has been trying to make use of the blessing of nature; on the other hand searched to protect against its negative effects. In many parts of the world, a great variety of buildings were constructed for the same primary purpose to protect and insulate people from ruthless conditions of nature. Insulation has been still quite important for human life safety beyond sheltering, comfort, health needs of human given the fact that the buildings and people face to destructive external factors nowadays [6].

On the other hand, insulation is quite relevant to energy efficiency. Developed and ever-developing countries need to more energy in proportion to their population. Renewable energy resources and energy conservation have gained importance to supply the needs of energy and to recover from external dependence in recent years. Turkey is dependent on outside financial sources with a ratio of 75 % in energy sector. Energy import is 55 billion USD/year and has the largest part of current account deficit of Turkey. It is indicated that energy consuming per year is nearly 75 billion USD in Turkey; moreover, 35 % of total energy (nearly 26.5 billion USD) is used in the buildings (Figure 1.1) [2].

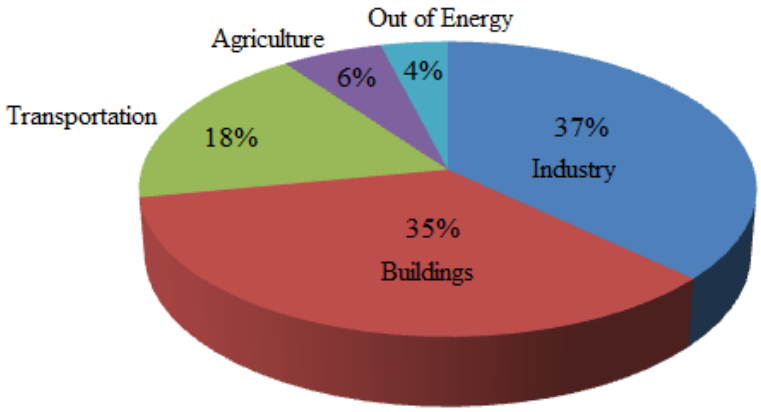


Figure 1.1. Energy consumption in Turkey [2].

In addition, 65 % of total energy used in the buildings is consumed for heating, cooling and ventilation of buildings and it consists of nearly 17 billion USD (Figure 1.2). It is also specified that 50 % of energy efficiency can be achieved with a proper thermal insulation, thus 8.5 billion USD energy saving can be obtained [2].

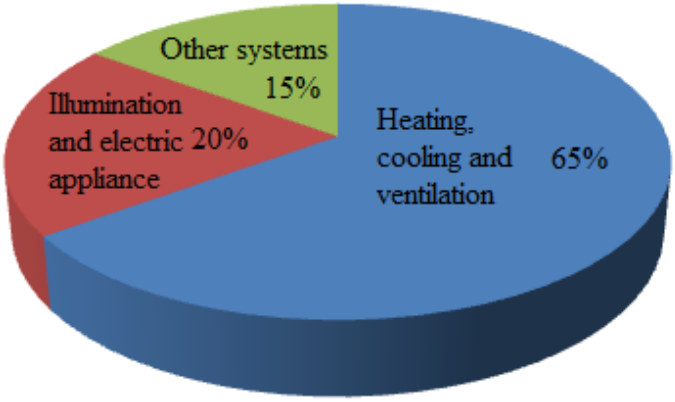


Figure 1.2. The ratio of energy consumption in buildings [2].

However, 85 % of buildings in Turkey have not been thermally insulated yet. Insulated buildings ratio of from 15 % to 26 % has been aimed until 2018 according to the 10th Development Plan of Turkish Prime Ministry [2].

Because of the importance of thermal insulation on energy efficiency, especially in the buildings, new generation thermal insulation materials are drawn attention to the heat insulation sector.

1.1.1. Thermal insulation

Thermal insulation means that the processes to decrease the heat transmission between two environments which have different temperatures. It involves some prevention measures like reducing the heat transfer at exterior facades, window glass and profiles, roofs, furniture and wirings of buildings to decrease the energy consumption for heating or cooling.

People should live in an environment at which temperature between 20-22°C need to be maintained for living in comfort. The heat, as a natural rule, transfers from warm to cool environment. The interior heat moves to outer environment at winter season; otherwise, the outer heat moves into the building at summer season. This heat transfer cannot be stopped but can be controlled by thermal insulation. Generally, the most of the heat loss occurs at the exterior walls in the buildings (Figure 1.3) [2].

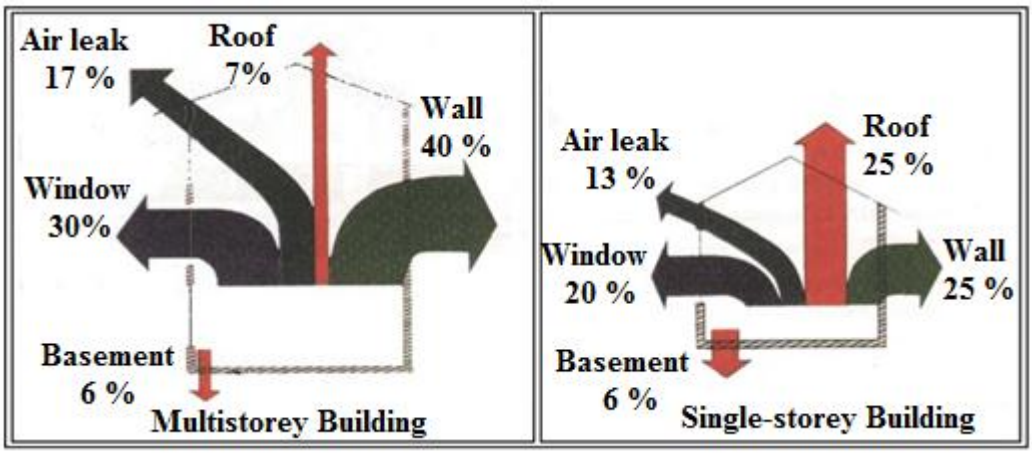


Figure 1.3. Heat losses at multi and single storey buildings [2].

Thermal insulation is not applied for only to prevent from cold, it is important also to be prevented from warm/hot weather. It is indicated that cooling cost in summer is higher than heating cost in winter. According to the standard "*TS 825 - Thermal insulation requirements for buildings"* [7] thickness of the thermal insulation material is very important. For example, thermal insulation materials used in cold climates should be thicker; on the other hand, thickness of the thermal insulation materials used in warm climates can be thinner [2]. This is because of the (U) value which depends on the thickness of the insulation material.

1.1.2. Thermal transmittance (U-value)

Thermal performance is measured in terms of heat loss, and is commonly expressed in the construction industry as a U-value or R-value. Thermal transmittance, also known as U-value, is the rate of transfer of heat through a structure (which can be a single material or a composite), divided by the difference in temperature across that structure (W/m^2K) [8]. U value indicates the level of insulation and varies according to the coefficient of thermal conductivity (λ), resistance of thermal insulation (R-value, m^2K/W) and thickness (m) of an insulation material.

It is a common knowledge that U-value should be lower for better insulation in a structure. If insulation is fitted poorly, with gaps and cold bridges, then the thermal transmittance can be considerably higher than desired. Thermal transmittance takes heat loss due to conduction, convection and radiation into account [8]. Calculation of basic U-value is also indicated in the standard TS 825 [7]. U-value can be measured by

finding the reciprocal of the sum of the thermal resistances of the material constituent of the building element. It is specified that as well as the material resistances, *the internal and external faces* also have resistance and these are fixed values which should be added at calculation [8]. Fix values of the resistances of the internal and external faces are indicated in the standard TS 825, and they are illustrated below in Table 1.1 and Figure 1.4.

Table 1.1. Calculated values of resistances of the internal and external faces [7].

Number	**Type of construction material**	**Resistances of the faces of the building***	
		R_i (m^2K/W)	**R_e (m^2K/W)**
1	External wall	0.13	0.04
2	External walls with dressing front, low walls separating the attic without heat insulation	0.13	0.08
3	Separating walls between the apartments, ladder wall, separating walls of working rooms having different usage purpose, partition wall adjoining the non-heated places, the lower adjacent to the space between the heat-insulated roof wall	0.13	**
4	Earth contact outer wall	0.13	0
5	A horizontal or inclined, which forms the boundary with the outside air of a living space, (non-ventilated roof) ceiling or roof above	0.13	0.04
6	Roof under an unused ceiling or ventilated area	0.13	0.08
7	Separating floor between apartments or between different usage purpose study rooms		
7.1	In the case of heat flow from top to bottom	0.13	**
7.2	In the case of heat flow from top to bottom	0.17	**
8	Basement ceiling	0.17	**
9	Bottom bases forming the boundary with the outside air of a living space	0.17	0.04
10	A basin sitting on the floor of a living space without a basement underneath	0.17	0

* To simplify, R_i =0.13 m^2K/W value can be used in all situation, and Re = 0.04 m^2K/W can be used in all situation except the situations number 4 and 10.

** Internal and external face thermal insulation resistance values should be same when construction material is indoor space.

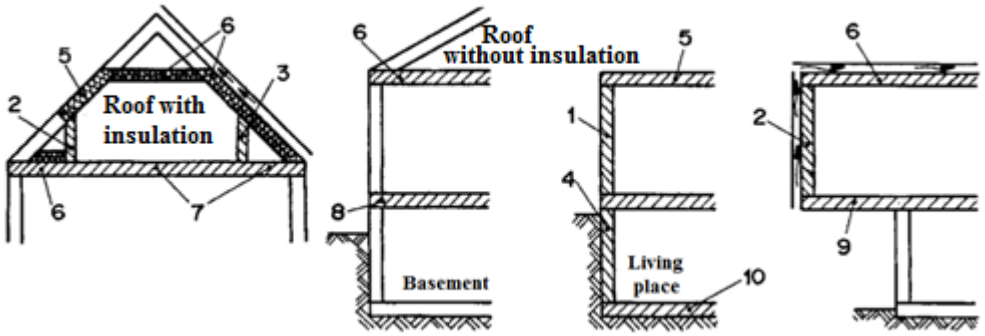


Figure 1.4. Design and places of construction elements [7].

Thermal transmittance (U-value) and thermal insulation resistance (R-value) values of a material are calculated according to the following equations (1.1, 1.2 and 1.3):

$$R = d/\lambda_c \quad (1.1)$$

$$R_{total} = R_i + R + R_e \quad (1.2)$$

$$U = 1/R_{total} = 1/(R_i + R + R_e) \quad (1.3)$$

R : Thermal insulation resistance of the construction material (m^2K/W)

d : Thickness of the construction material (m)

λ_c : Calculated value of coefficient of the thermal conductivity (W/mK)

R_{total} : Total thermal insulation resistance value (m^2K/W)

R_i : Thermal insulation resistance of the internal face (m^2K/W)

R_e : Thermal insulation resistance of the external face (m^2K/W)

U : Thermal transmittance value (W/m^2K)

1.1.3. Thermal insulation materials

To classify a material as a thermal insulation material, coefficient of thermal conductivity must be lower than 0.065 W/mK according to the ISO and CEN standards. In general, the most considered properties for an insulation materials are coefficient of

thermal conductivity (W/mK), density (kg/m^3), fire class, temperature range for usage area (°C), mechanical strength (MPa), water vapor diffusion resistance (μ), water absorption (%) and dimensional stability [6].

Thermal conductivity is a property to distinguish a thermal insulation material from each other. It is a general knowledge that porous and low density materials have low thermal conductivity. Also, the smaller the coefficient of thermal conductivity value, the higher the resistance to heat transfers. In the standard TS 825 [7], calculated thermal conductivity (λ) values for several insulation materials were indicated according to their densities. It is specified that in the standard TS 825, low density is also a significant property to provide heat insulation.

Another important property of thermal insulation materials is the coefficient of water vapor diffusion resistance (μ). The water vapor occurred at interior environment have a potential to harm the buildings. The water vapor passes from inside of pores of construction element and tries to arrive outer environment. During this transmission inside the construction element, a part of vapor condenses and transforms into water when it saturates or contacts with a cooler surface. This condensation is an unwanted situation because of the damaging effect on concrete and fittings [2]. For this reason, insulation materials are preferred with a high μ value.

In Turkey, commonly used thermal insulation materials are extruded polystyrene (XPS), expanded polystyrene (EPS), fiber glass, stone wool, polyurethane foam (PUR), phenol foam (PF), expanded perlite boards (EPB) and expanded cork boards (ECB). Technical properties of these popular and conventional thermal insulation materials were given in Table 1.2 and compared with foam glass according to the commercial information. Illustration of these thermal insulation materials were also given in Figure 1.5.

Table 1.2. Comparison of foam glass and other thermal insulation materials used in Turkish market [2, 7, 9-24].

Type of Commercial Insulating Boards	Thermal Conductivity (W/mK)	Density (kg/m^3)	Compressive Strength (MPa)	Water Absorption* (%)	Vapor Diffusion Resistance (μ)	Fire Class	Temperature Range for Usage Area (°C)	Dimensional Stability
XPS	0.030 – 0.035	24 - 37	0.1 - 1	0.7 - 3	80 - 250	E	-50 to +70	Stable
EPS	0.030 – 0.038	16 - 30	0.03 – 0.5	1 - 5	20 - 100	E	-	Stable
Fiber Glass	≤0.040	40 - 120	0.0005 – 0.5	3 - 10	1	A1	-50 to +250	Stable
Stone Wool	0.035 – 0.040	30 - 200	0.0005 – 0.5	9 - 12	1	A1	-50 to +600	Stable
PUR	0.025 – 0.040	40	>0.095	2	30 - 100	D/E/F	-200 to +110	Stable
PF	0.030 – 0.045	35 - 120	0.10 – 0.15	9	7 - 10	B1	-180 to +120	Stable
EPB	0.045 – 0.065	50	0.31	6	5	A	-250 to + 1000	-
ECB	0.045 – 0.055	80 - 500	-	-	5 - 10	E	-180 to +100	-
Foam Glass	0.045 – 0.060	100 - 160	≥0.4 - 3	<0.2	>40000	A1	-268 to +482	Stable

*Long term water absorption by immersion

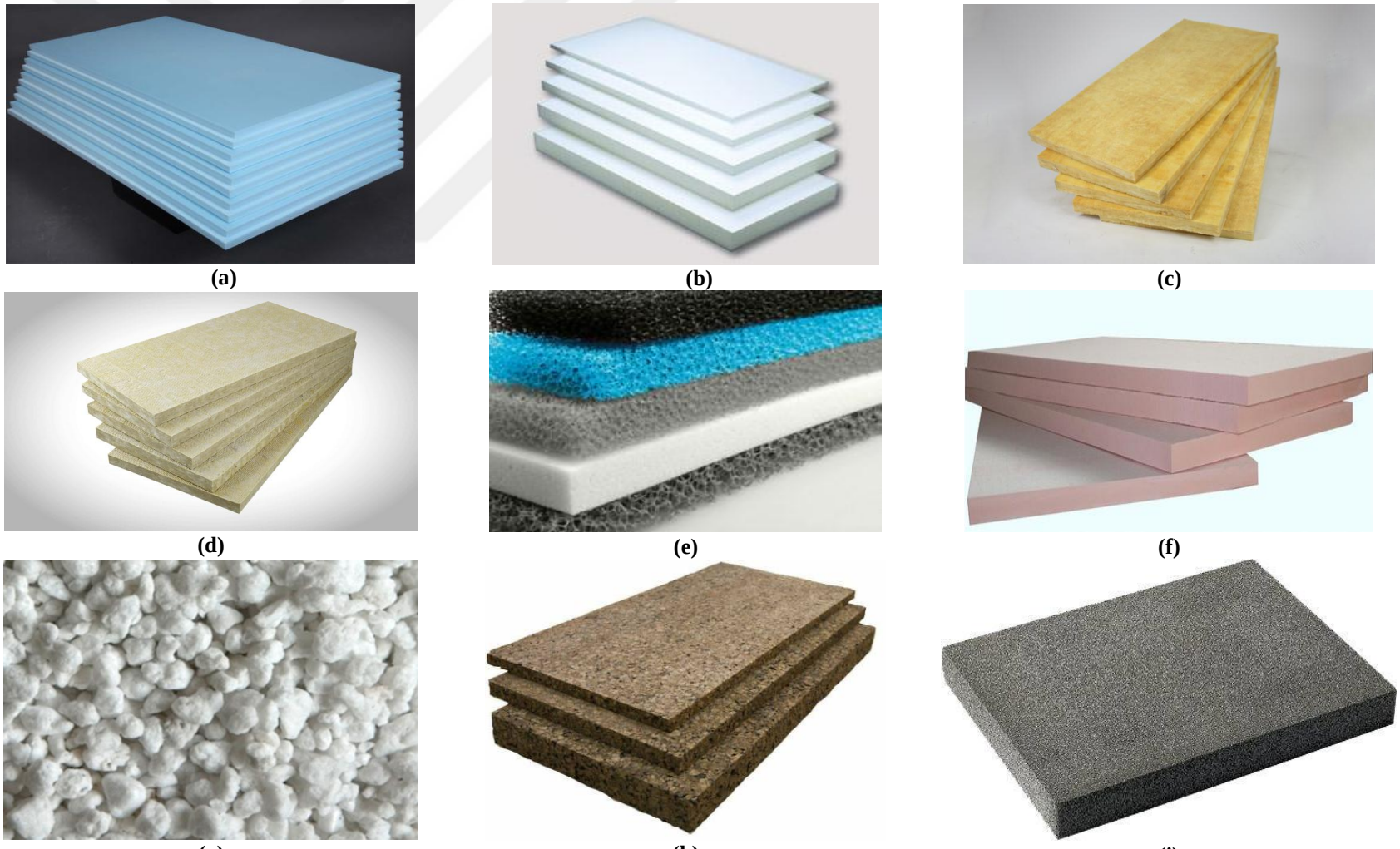


Figure 1.5. Illustration of thermal insulation materials used in Turkey; **(a)** extruded polystyrene (XPS), **(b)** expanded polystyrene (EPS), **(c)** Fiber glass, **(d)** stone wool, **(e)** polyurethane foam (PUR), **(f)** phenol foam (PF), **(g)** expanded perlite boards (EPB), **(h)** expanded cork boards (ECB) and **(i)** foam glass [9-13, 20, 21, 25-27].

1.2. Foam Glass

1.2.1. History of foam glass

The foam glass has been produced since 1930's. The first inventors of foam glass are uncertain because of that lots of the patents granted in the same period of time. In 1931, foam glass production method was developed [5] by using silica together with 20 % combustible material (lignite, coal and wood) and foaming agent (hydrochloric acid, sodium hydrochloride solution), then, porous product was obtained by sintering this mixture at 1500°C. In another study [5] conducted in 1932, a different production method was performed in Mendeleev Institute, Moscow. Foam glass was achieved with heating of finely ground glass particles and calcium carbonate at 850°C.

Another technology developed in 1934 for foam glass production from finely ground silica, borax and zinc oxide mixture. In the later years, this technology was improved by Pittsburgh Plate Glass & Corning Glass Works (Pennsylvania, USA) and foam glass manufacturing patent was obtained with using additionally foaming agents (like air, water vapor) in 1940. The product, foam glass was easily found a place in the new insulation materials market with its excellent properties like lightweight, rigid, resistance to fire, air, rodent and insects, and mass production was begun in the USA, in 1943 [5].

The early products manufactured in 1960's were blocks and preshaped objects, and producers were using specially formulated glasses to obtain foam glass products like alumina-borosilicate glass. In recent years, however, the main foam glass producers in Europe and North America use high percentage (nearly 98 %) of post-consumer waste glass in their products [28].

1.2.2. Definition of foam glass

Foam glass is mainly signified as cellular glass. It is generally produced by using starting glass and gas-generating agent (mostly named as foaming agent). There are two fundamental foam glass production processes according to the related patents. First one is based on the production of foam glass by sintering of fine glass powders-foaming agent mixture, and the second one is achieved by directly adding some fluids and

foaming agent (CO_2, air and water vapor) into molten glass. The foam glass can be either made from molten glass or sintered glass particles. Recently, pure glass with specific compositions has been used to produce foam glasses; however, post-consumer waste glass with a high percentage has also been used by many foam glass producers in Europe and North America. According to recent works, glass powder, foaming agent and optionally mineral additives are mixed and ground together. The mixture is shaped usually by press or filled in a mold, then heated to elevated temperatures. At this stage, glass particles soften and gas bubbles evolved from the foaming agents by sintering reactions. When these gas bubbles are obtained, they remain within the glass which is driving force for viscous flow. These gas bubbles, which are preliminarily small and spherical, cause an increase on gas pressure and expansion of glass. At the end of the process, glass foam having polyhedral cells is obtained [5, 28]. Foam glass is composed of incomparable properties: lightweight, rigid structure, thermally insulating, compressive strength, nonflammable, freeze tolerant, nontoxic and chemically inert, rodent and insect resistant, bacteria resistant, water and steam resistant. These properties supply a reliable material for structural and health aspects beside thermal insulation and fireproofing. In addition, all these combination of properties provide that different application area like roof, wall, floor and ceiling under hot or cold conditions. Foam glass products can be easily cut and shaped, thus they can be produced in any desired shape [5]. Figure 1.6 illustrates some examples for foam glass products [29, 30].



Figure 1.6. Illustration of some foam glass products [29, 30].

1.2.3. Properties of foam glass

Technical properties of foam glass material are defined in the standards TS EN 13167:2013-04 [24] and BS EN 13167:2012+A1:2015 [31] “Thermal insulation

products for buildings — Factory made cellular glass (CG) products— Specification". According to these standards, required properties of foam glass material are illustrated in Table 1.3. Also, physical properties declared by Pittsburgh Corning UK Ltd., one of the main producers of foam glass in the world, are given in Table 1.3.

Table 1.3. Physical properties of foam glass [24, 31-54].

Physical property		**Measurement method**	**TS EN 13167:2013-04 (SI value)**	**Pittsburgh Corning Foam glass (SI value)**
Thermal conductivity (W/mK)		ASTM C518 ASTM C177 ISO 13787	0.045 – 0.060	0.039 – 0.050
Compressive strength (MPa)		ASTM C165 ASTM C240 ASTM C552 EN 826	≥0.4 - 3	0.62
Bending strength (MPa)		ASTM C203 EN 12089	≥0.2 – 1.4	0.48
Density (kg/m^3)		TS EN 1602	90 – 180	100 - 160
Short term water absorption by partial immersion (kg/m^2)		TS EN 1609	≤0.5	≤0.5
Long term water absorption	Partial immersion (kg/m^2)	TS EN 12087	<0.5	-
	Total immersion (%)		-	<0.2
Water vapor diffusion resistance (μ)		ASTM E96 EN 12086 EN ISO 12572	>40,000	∞
Flammability and reaction to fire		ASTM E84 EN ISO 1182	A1	A1
Dimensional stability (%)		ASTM C411 EN 1604	Length: 0.5 Thickness: 1	Excellent - Does not shrink, swell or warp
Coefficient of thermal expansion (K^{-1})		ASTM E228 BS EN 13471	8 x 10^{-6} K^{-1} to 10 x 10^{-6} K^{-1} at 23°C	9.0 x 10^{-6} K^{-1} at 25 to 300°C



(a) Water-proof **(b)** Pest-proof **(c)** Compression-proof

(d) Incombustible **(e)** Dimensionally stable **(f)** Vapour-tight

(g) Acid-resistant **(h)** Easy to work with **(i)** Ecological

Figure 1.7. Illustration of required properties of foam glass [55].

Some examples for foam glass products are shown in Figure 1.7. According to the related standard and commercially sold foam glass material, there is no water and moisture absorption in the structure because of its primary raw material is glass. Resulting from inorganic material content, insect and pest proof are ensured naturally. Cell geometry within the porous structure provides satisfactory compressive strength. Foam glass is a fire proof material because of its pure glass content in high percentage. Fire resistance property supplies a natural protection in the event of fire. In addition, homogeneous glassy cells within the structure prevent transportation of vapor. This prevention maintains constant thermal insulation properties for many years. Foam glass is dimensionally stable material, because of the high glass content which renders no shrinkage or swelling. Dimensional stability is resulting from having low thermal expansion coefficient value. Another important property is acid resistance; aggressive media and atmosphere cannot destruct the insulation material because of foam glass provides a resistance to acids and organic solvent. Easily cutting (by saw blade or hand

saw) and working are other advantages to use foam glass in any required dimensions. Also, it can be produced for using in various application areas like pipe, walls, floor and roof etc. Foam glass is an environment friendly material. It does not contain any eco-toxic components; therefore it can be recycled and reused after using as an insulation material [55].

1.2.4. Application area of foam glass

The main application areas of foam glass material are given below and illustrated in Figure 1.8:

- Base and roof insulation
- Interior insulation for floor and walls
- Structural blocks
- Concrete production as loose aggregate
- Between cavity walls, bricks and briquette blanks
- Tank and pipe insulation
- Underground systems
- High and low temperature applications
- Chemical storage tanks due to its high corrosion resistance
- Dust suppression, filtration and as filler material [28, 56, 57].



Figure 1.8. Some examples for foam glass applications [56, 57].

1.3. Foam Glass Production Process

The main principle of foam glass production is releasing of gas bubbles within the glass at a temperature between 700-900°C. Molten glass or sintered glass particles can be used for production. The main stages of production are mixing the foaming agent and glass particles, and then foaming reaction and gas generation occur at elevated temperature. Expansion of gas within viscous molten glass causes a porous structure with close cells. There are three different types of foam glass production process. Illustration of these foam glass products were given in Figure 1.9 [28].

- Continuous sheet process to produce loose foam glass aggregate
- Continuous process to produce foam glass blocks and shapes
- Foam glass pellets process

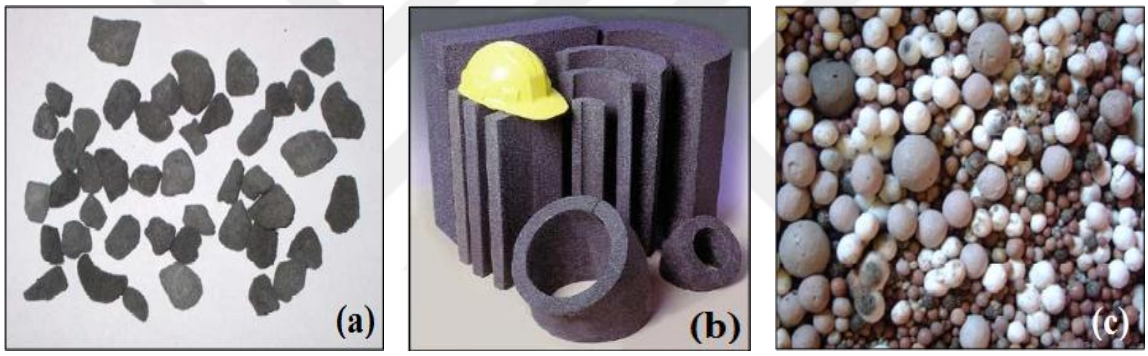


Figure 1.9. (a) Loose aggregates, **(b)** foam glass blocks and **(c)** foam glass pellets [28].

1.3.1. Continuous sheet process for loose foam glass aggregates

Waste glass is the main raw material in this process. Because of the initially obtained waste glass (container and/or flat glass) size range is between 5-20 mm, the glass cullet is ground in two steps by using hammer mill and ball mill to gain fine glass powders. In the continuous process, fine-ground glass powders and foaming agent are mixed and placed on a moving belt. This mixture on the moving belt is fed into the furnace and heated at 700-900°C. To achieve continuous production, the feedstock is continuously fed into moving belt that transfers through the furnace. After obtaining foam glass as sheet form, the foam glass slabs can be broken up into desired particle ranges to have loose aggregate. Figure 1.10 shows foam glass sheet production with continuous furnace and obtaining of loose aggregates in a foam glass facility, Misapor AG,

Switzerland. The most important advantage of loose aggregate is signified as lightweight nature. Lightweight structural concrete can be produced by using loose foam glass aggregate. Also, it is indicated that loose foam glass aggregate can be used in ground stabilization and water drainage systems [28].



(a) **(b)**

Figure 1.10. (a) Foam glass sheet furnace, **(b)** production of loose aggregate [28].

1.3.2. Continuous process for foam glass blocks and different shapes

This process has quite similar technique to previously stated sheet production process except that the blocks and different shapes are achieved rather than a continuous sheet. According to the commercial production process conducted by Pittsburg Corning UK Limited [32], glass which has desired composition is used in the production. During the process, raw materials (recycled glass, feldspar, sodium carbonate, iron oxide, sand, sodium sulphate and sodium nitrate etc.) are mixed and melted in the furnace at a constant temperature of 1250°C. Molten glass is removed from the furnace, cooled and then ball milled to have fine powder. After then, foaming agent (usually carbon black) is added. The glass and foaming agent mixture is filled into stainless steel mold. The filled mold continues through the furnace which has annealing and cooling zone. Annealing zone provides controlling of cooling and prevents the internal strain and thermal stress, thus foam glass product having suitable form can be produced. Finally, the blocks are cut into desired size and shapes [55]. Figure 1.11 represents molten glass and foam glass production within the stainless steel mold.



(a) **(b)**

Figure 1.11. (a) Molten glass and **(b)** foam glass block production within mold [32, 58].

1.3.3. Spherical pellet production

Foam glass pellet production has a different method from the others. Fine glass powders and foaming agent are transformed into spherical shape pellets and fed into rotary furnace. After foaming reactions, foam glass pellets with spherical shapes are produced. Pellet production process and example of products are illustrated in Figure 1.12 [28].



(a) **(b)**

Figure 1.12. Foam glass pellets **(a)** production by rotary furnace and **(b)** products [28].

1.4. Important Parameters in Foam Glass Production

Various parameters which affect the foam glass properties were investigated in the literature. It was seen that the following factors were important to designate of foam glass properties: starting glass type and composition, foaming agent, foaming mechanism, heating rate, foaming (sintering) temperature, heating/holding time and cooling rate [5].

1.4.1. Glass

The primary raw material is waste glass (>66%) in the modern foam glass production [55], using of different types of recycled glass waste to obtain foam glass as a valuable insulation material were studied by various researchers. It was realized that sheet, soda-lime and borosilicate glass cullet [59-61] are mostly used as glass source in the foam glass production, followed by lead and barium silicate glass wastes from Cathode Ray Tube (CRT) [62] and fluorescent lamps waste glasses [63]. As indicated in the study conducted by Scarinci et al. [5], any change in the chemical composition of raw materials is quite effective on viscosity and foaming temperature. Thus, sintering schedule of foam glass is dependent on the glass source chemical composition also. Since the foam glass is mostly composed of glass, different types of glass sources create different foam glass structures and properties.

The following factors are important when choosing glass cullet:

- Softening temperature of glass should be low, thus the generated gas can stay within the viscous glass during foaming. Otherwise, gas escapes from the body and there will be inhomogeneous and non-porous structure.
- At sintering temperature, viscosity change rate of glass should be low. Thus, more homogenous cell sizes can be obtained.
- Thermal expansion coefficient of glass should be low. This feature reduces annealing and cooling time.
- Chemical durability of glass should be high. Foam glass products are used for hard conditions, so chemical durability is a necessary property.
- Vitrification tendency of glass should be low, because vitrification decreases the strength of the product.

In the Table 1.4, chemical compositions of some waste glasses which are used in foam glass production are given based on the papers in the literature. It was seen that silica (SiO_2) ratio of sheet and soda-lime glass is higher than other CRT and fluorescent based waste glass. Also, it can be said that using of CRT and fluorescent based glass wastes requires higher caution because of including heavy metals (PbO and BaO) in high percentage. Thus, one of the reasons to prefer soda-lime based glass wastes rather than CRT and fluorescent glasses; they are being healthier and environmentally friendly.

Table 1.4. Comparison of the chemical compositions of some waste glasses from the literature [59, 63-66].

Oxide (wt. %)	Tulyaganov et al. Sheet Glass	Bernardo et al. Soda Lime Glass	Méar et al. Funnel Glass from CRT	König et al. Panel Glass from CRT	Mugoni et al. Fluorescent Lamp Glass
SiO_2	70.64	70.8	52	60.1	67.90
Al_2O_3	0.68	2.4	4	2.5	2.26
Fe_2O_3	0.18	0.3	0.06	-	0.08
CaO	9.93	9.4	3.8	-	5.09
MgO	3.55	2.1	1.8	-	2.96
Na_2O	13.66	13.0	6.8	7.9	17.50
K_2O	0.29	1.1	7.8	7.1	1.60
SO_3	0.21	0.09	-	-	-
BaO	-	0.2	1	9.5	0.95
TiO_2	-	0.1	0.05	-	-
ZrO_2	-	0.1	-	1.9	-
B_2O_3	-	0.12	-	-	-
PbO	-	0.07	22	0.05	0.78
ZnO	-	0.12	-	-	-
As_2O_3	-	0.02	0.01	-	-
Sb_2O_3	-	0.01	0.25	-	0.08
SrO	-	-	0.5	8.2	0.07
F	-	0.08	-	-	-
Others	-	-	-	2.8	0.74

1.4.2. Foaming agents

Scarinci et al. (2005) reported that any glass (favorably in powder form) can be transformed into foam by addition of suitable foaming agents. Based on the gas generation mechanism, foaming agents are classified into two types: reducing and decomposable agents [5].

Gas generation occurs from reaction or decomposition at above glass softening temperature (corresponding to a viscosity of $10^{6.6}$ Pa.s). If gas evolution occurs at temperatures below the softening point of glass, gaseous products cannot be kept by the mass because of the glass powder is not sintered yet to form closed porosity. In

addition, if the gases arise when the glass viscosity is too low, evolved gases escape from the melted body forming a non-porous glassy product. It was also stated that most proper viscosity of standard sheet soda-lime glass composition is between 800-1000°C to achieve maximum porosity and minimum apparent density of foam glass. The viscosity and foaming temperature are very sensitive to foaming agent type and amount. The same foaming agent type can differently affect the property and structure of different glasses; also, the same glass can be differently affected by addition of different foaming agents. Moreover, quantity of foaming agent is very influential on the microstructure and size of final pores. Thus, it was emphasized that foaming agent type and addition ratio should be chosen carefully because these factors are quite important to create foam glass structure [5].

1.4.2.1. Reducing agents

Reducing agents, also known as redox agents are usually carbon-based materials like carbon (C), coke, anthracite, soot, graphite and silicon carbide (SiC) which react with glass and form gaseous products of CO_2 or CO [67].

SiC as reducing - foaming agent

Silicon carbide was highly investigated as foaming agent by many researchers in recent years. In a detailed study conducted by Brusatin et al. (2004), SiC was discussed as extremely effective reducing agent to provide the possibility of control and variability of microstructure [68]. It was also reported that one of the reason of preferring SiC as a foaming agent is controlling ability of the microstructure [28]. When silicon carbide is mixed with glass powder in various composition ranges, it causes foaming of glass by generating of gas bubbles due to oxidation (redox) reactions. Reaction temperature was specified to be higher (≈1000°C) when comparing with other foaming agents (carbonates, carbon and organic compounds) [68].

According to the study of Tulyaganov et al. (2006), even very small additions of SiC can influence the foaming process by oxidation and releasing CO_2 during heat treatment [59]. In another study conducted by Bernardo et al. (2007), it was shown that increasing the amount of SiC-based waste in soda-lime glass caused decreasing of the relative density of foam glass products [64]. Because of providing the homogeneity of foam

glass, it was demonstrated that compressive and bending strength was strongly influenced by SiC addition ratio. The primary reason for this behavior is related to gas generating effect of SiC addition. When SiC addition ratio is low, gas evolution and expansion rate is also low. Thus, higher strength can be achieved in glass foam because of the solid structure in the sample is more dominant rather than porous structure [69]. There is an example from the study conducted by Méar et al. (2006) to show the foaming agent addition ratio effect on pore size and microstructure which is shown in Figure 1.13. It was reported that pore size was quite important factor on density, mechanical strength and thermal conductivity which was increased with higher foaming agent addition [65].

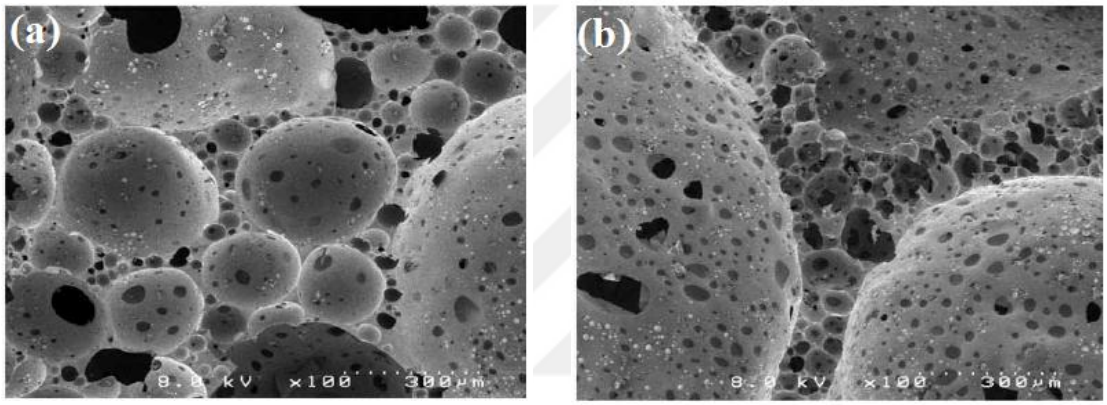


Figure 1.13. Microstructures of foam glass samples produced from funnel glass with addition of **(a)** 1 wt. % SiC and **(b)** 5 wt. % SiC at same sintering schedule.

1.4.2.2. Decomposable agents

On the other hand, decomposable agents, also known as neutralization agents are generally carbonates like $CaCO_3$, $MgCO_3$ and Na_2CO_3 or organic compounds which decompose upon heating and release gaseous products. Also, it was specified that the reaction products such as CaO decomposed from $CaCO_3$, remain in the glass and enhance the glass properties like viscosity and crystallization behavior [70].

1.4.3. Foaming mechanism

The basic mechanism of foaming in foam glass is reported in the related literature that transformation of glass powders into a viscous liquid occurs between 700-900°C and then the gas is released by decomposing or oxidizing of foaming agent into bubble

forms. The important thing is that glass should have sufficient viscosity not to allow the gas bubble to rise through the mass of the body but remain in position as entrapped bubbles during the foaming [28]. To achieve successful foaming, optimum thermal treatment schedule should be applied according to glass viscosity and foaming agent reactions [68]. Foaming agents principally determine the foaming mechanisms which are foaming by reaction and foaming by thermal decomposition.

1.4.3.1. Foaming by reaction

It was reported in many study [5, 59-60, 68, 71] that glass foam can be produced by chemical reaction of suitable foaming agents with the atmosphere and the glass above the softening point. In general, carbon dioxide is released within the softened glass body by oxidation of carbon-based foaming agents like pure carbon, SiC, sugar or organic wastes. Usually a mixture of CO_2 and CO constitutes the present gas in the closed pores.

Foaming cannot be considered as only evolution of gas bubbles in a softened glass, it also depends on physicochemical interactions in the glass on gas releasing. As a result of reducing reactions, a certain amount of residual oxides stay behind to be dissolved in the glass. Thus, CO/CO_2 formation by reduction of oxides in the softened glass modifies the chemical composition and viscosity of the system.

Silicon carbide (SiC) is mostly preferred as very effective foaming agent because it provides homogenous, controlled and precise cell sizes in the glass foam [5]. In a compacted glass-SiC body, the desired oxygen for the foaming process can be achieved from two different sources: (a) the oxygen dissolved in the glass and (b) the furnace atmosphere [71].

It was reported that SiC requires higher temperature (950-1150°C) than carbon for foaming process, also it was indicated that the reactions of SiC with the atmosphere and glass are much more complex. The thermodynamically possible reactions between SiC and the gas in various atmospheres were given in Table 1.5. After these reactions, a certain amount of silicon containing and carbonaceous solid or gas products occur. These carbonaceous matters provide foaming; on the other hand, residual silicon oxide combines with glass. Released silicon oxides from foaming agent may precipitate as cristobalite and result in partial crystallization of glass [5].

Table 1.5. Possible reactions between SiC and gas species in various atmospheres [5].

Reaction	ΔH/kJ (at 727°C)	ΔH/kJ (at 1227°C)
$SiC + ½ O_2 \rightarrow SiO + C$	-134.7	-179.08
$SiC + ½ O_2 \rightarrow CO + Si$	-157.3	-204.60
$SiC + O_2 \rightarrow SiO + CO$	-333.5	-412.3
$SiC + O_2 \rightarrow SiO_2 + C$	-656.1	-575.7
$SiC + O_2 \rightarrow CO_2 + Si$	-350.6	-357.3
$SiC + 2O_2 \rightarrow SiO_2 + CO_2$	-1048.1	-970.7
$SiC + 2CO \rightarrow SiO_2 + 3C$	-258.6	-91.2
$SiC + 3H_2O \rightarrow SiO_2 + CO + 3H_2$	-279.9	-327.2
$SiC + 4H_2O \rightarrow SiO_2 + CO_2 + 4H_2$	-281.6	-316.3

1.4.3.2. Foaming by thermal decomposition

Foaming process by thermal decomposition is conducted by using carbonates like $CaCO_3$ and Na_2CO_3. After decomposition agents are added into glass powders, the glass and carbonate mixture are heated to soften the glass. At this stage, the calcium or sodium carbonate particles decompose to the oxide with simultaneous generating of gaseous carbon dioxide as shown in the Equation 1.4 below.

$$CaCO_3 \rightarrow CaO + CO_2 \quad (1.4)$$

Similar to the foaming by reaction, the residual oxide gets involved to the molten glass mass and acts a glass modifier, thus altering the viscosity of the molten glass. On the other hand, the releasing CO_2 gas is trapped in the viscous glass and force the molten glass mass to expand by its continuously increasing pressure. Finally, porous structure is achieved after cooling molten glass and decomposable agent mixture [5].

1.4.4. Heating rate

Heating rate is defined by many sources [5, 28, 72] as one of the most effective factors to be controlled for obtaining foam glass product. It was indicated that heating rate to foaming temperature must be at slow rate because foam glass mixture has low thermal conductivity. To supply uniform temperature distribution, heating rate of larger samples should also be slower. Rapid heating (i.e. 40°C/min) can cause large cracks through the

glass mass, on the other side, slow heating can derive to early release of gas from the foaming agent before achieving the sufficient viscosity of glass (low enough to allow the glass to expand). Thus, it was also defined that heating rates of 5-10°C/min are usually suitable [5, 28].

1.4.5. Foaming temperature

Foaming temperature and glass viscosity (hardly dependent on temperature) are quite related. When applied foaming temperature is too high, the melt viscosity will be too low (i.e. $<10^3$ Pa.s) and controlling of the structure is difficult, bubbles will rise from the top of the mold and the body will collapse or shape-distorted with non-uniform bubble distribution may occur. Thus, foam glass body cannot be achieved. On the other side, if the foaming temperature is too low, the glass viscosity will be too high. Thereby, foaming will be difficult and little volume expansion will occur. Finally, separating walls (struts) between the cells do not complete and open porosity which cause high water absorption will be generated because of low foaming temperature [5, 28]. Thus, foaming temperature should be just above the glass softening point; therefore, generating gaseous can be hold by viscous glass melt without escaping from the body and porous structure can be achieved with closed cells. Foaming temperature effect on the microstructures of foam glass was shown in the study [65] conducted by Méar et al. (2006). Foam glass samples were produced at 750, 850 and 950°C with using 5 wt. % SiC foaming agent and 95 wt. % funnel glass. Microstructure results of these samples at different foaming temperatures were given in Figure 1.14. As it can be seen in the Figure 1.14, pore diameters increased with increasing of foaming temperature. The study explained that higher temperatures above 900°C caused tearing of the struts of the pores [65].

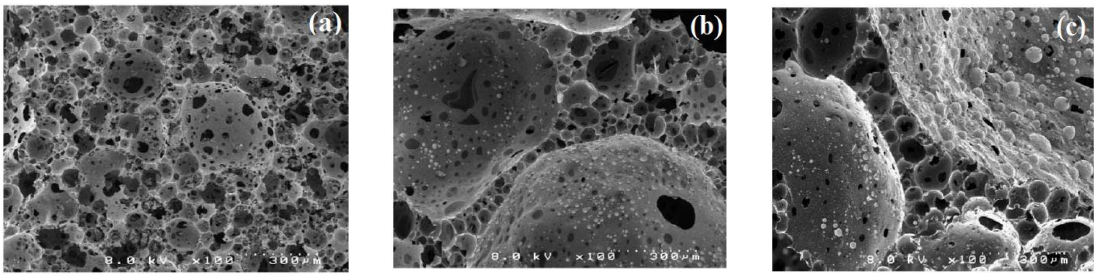


Figure 1.14. Foam glass samples produced at **(a)** 750°C, **(b)** 850°C and **(c)** 950°C [65].

1.4.6. Heating time

Heat treatment time or holding time is quite effective on the density of foam glass. The density declines down to the minimum value during gas releasing. When gas evolving finishes, distortion and collapse of the foam cells by integration or merging of pores begins due to high gas pressure inside of the pores. After then, an increase in density proceeds. For this reason, a proper holding time must be calculated and foam glass should be removed from the hot zone before the distortion of pores and increasing density. Figure 1.15 shows the relationship between heating time and density of foam glass [5]. Moreover, it was notified that increasing in reaction/holding time causes an increase in pore size of foam glasses [65]. This indication conforms to the decreasing apparent density with heating or holding time as depicted in Fig. 1.15.

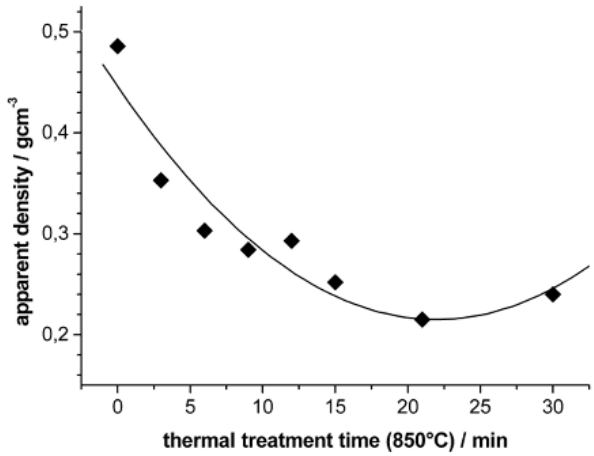


Figure 1.15. Apparent density of foam glass as a function of thermal treatment time [5].

1.4.7. Cooling rate

It is suggested that slow cooling rate (adopted glass transformation range) is essential to prevent residual stress generation in glass foam. While cooling process proceeds, the stress within the cells decreases, and some shrinkage and tensile stress between the layers can occur. Thus, rapid cooling can cause internal cracks due to residual stress generation [5].

1.4.8. Initial particle size of raw materials

Both of the glass and foaming agent initial particle sizes have significant relation with foam glass microstructure and pore size distribution. It was indicated that initial glass

powder size should be fine (<0.4 mm), otherwise, foaming process cannot be derived. Glass particle size effect on the microstructure and pore diameter of foam glass was shown in Figure 1.16. It was seen that coarser glass powders cause larger pores; on the other hand, fine glass powders provide smaller pore size which is essential for higher mechanical strength [5].

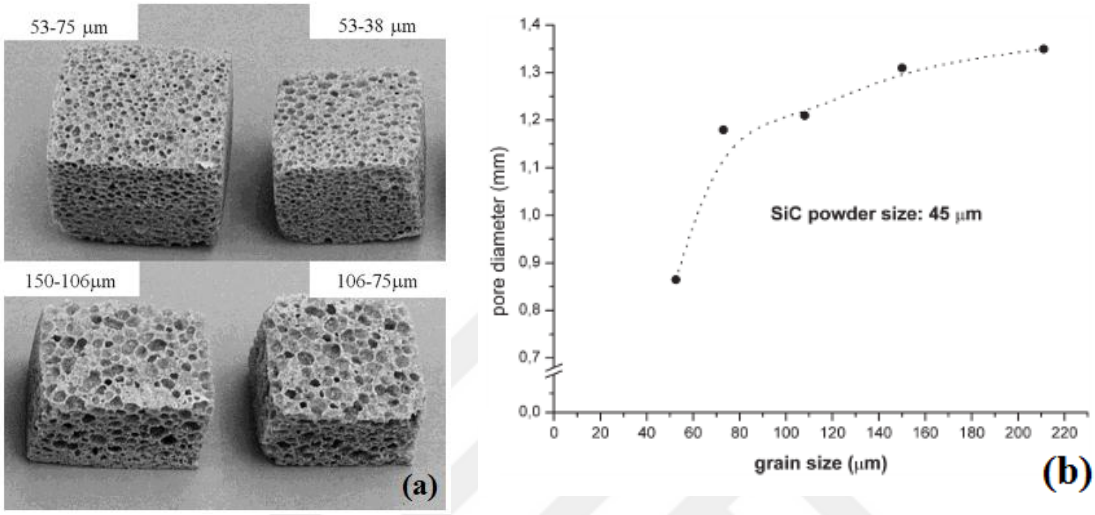


Figure 1.16. Effect of glass powder grain size on **(a)** microstructure, **(b)** pore diameter of foam glass [5, 68].

It is explained that smaller cell dimension and higher homogeneity are achieved because of using fine glass powder. Another explanation is that if the glass particle size is much coarser than foaming agent, there will be an agglomeration and separation inside the mixture because of larger voids among individual glass grains [68]. In addition, foaming agent particle size is quite effective on foaming process. It was noted by several researchers that when comparing coarser foaming agent (i.e. 74-78 μm SiC) addition, with fine foaming agent (i.e. 4-7 μm SiC) addition, remarkably higher volume expansion was obtained with finer foaming agent at lower sintering temperature. Another important factor is glass and foaming agent particle size relation. It was seen that more homogeneous microstructures can be achieved if initial particle sizes of glass and foaming agent's size are closer. When glass and foaming agent particle sizes have very different dimensions, homogeneity of microstructure is affected negatively [5, 64, 68]. It was also remarked that initial particle size of glass powders dominate the density, mechanical strength and thermal conductivity properties of foam glass. Fine glass powder supplies more successful foaming and large amount of cells which also results in lower density. In general, lower density provides lower thermal conductivity and

higher insulation properties. Moreover, because of the smaller cell size from fine glass powder, higher compressive strength is achieved [5]. It can be concluded that finer glass and foaming agent powders are essential to have desired foam glass properties.

1.5. Literature Survey

At the begining of the study, foam glass production methods were surveyed in detail from the literature. Especially similar studies in which waste glass and SiC containing mixture were summarised and the inferences from the literature were given below.

Tulyaganov et al. (2006) studied about production of high compressive foam glass with using two different glass cullet and SiC powder. In the study, sodium-calcium-silicate sheet glass was used as main raw material, while second glass source was alkali earth aluminosilicate glass. Before the mixture preparation, firstly sodium-calcium-silicate sheet glass was crushed by using crushing machine, then dry milled by using porcelain ball mills to have fine glass powder and coded as "C" glass. On the other hand, alkali earth aluminosilicate glass was produced by researchers with using natural raw materials like quartz, sand, calcite, magnesite and kaolin; moreover, $NH_4H_2PO_4$ and CaF_2 powders were also added as P_2O_5 and fluorine sources, respectively. These raw materials were mixed and melted in corundum crucibles in electric furnace at 1400-1420°C for 1 hour and alkali earth aluminosilicate glass frit was achived by quenching of glass melt. Then this glass frit was also dry milled to gain fine glass powder and named as "AD" glass. Mean particle sizes of C glass, AD glass and SiC powders were measured as follow: 9 μm, 13 μm and 17 μm, respectively. When properties of C and AD glass powders were analysed, coefficent of thermal expansion values were measured as $83.0x10^{-7}$ K^{-1} and $86.5x10^{-7}$ K^{-1}, transformation temperature values were measured as 540°C and 720°C and dilatometric softening points were measured as 590°C and 760°C, respectively.

Seven different batch composition was prepared by using different mixture ratios: 89-99.75 % C glass, 1-10 AD % glass and 0.25-1 % SiC. Batch mixing was applied for 30 minute by using cyclindrical rotary mixer. Mixtures were put in stainless steel moulds and sintered at air atmosphere with applying following regime: Heating up to 900°C (5°K/min), holding for 30 minutes at peak temperature, further heating up to 950°C (5°K/min) and holding for 30 minutes. When foam glass samples were obtained (Figure

1.17) they were analysed and observed that increasing of SiC addition from 0.25 % to 1 % caused more homogeneous microstructures. Beside, AD glass addition into C glass supplied decreasing of pore sizes. Increasing of AD glass amount provided a honeycomb structures with pentagonal and hexagonal like shaped pores in foam glass. Optimum products were achieved by using 1 % SiC and 5-10 % AD glass. At the end of the study, it was shown that foam glass production was possible with using recycled sheet glass and SiC as foaming agent. Final products satisfied the structural and insulating properties for building constructions. Alkali earth aluminosilicate glass powders enhanced the sintering and increased compressive strength (2.132-2.601 MPa) as a result of occuring honeycomb pore structure. Also, high amount of porosity was obtained with a range of 87.90-90.05 % with low bulk density values as 0.252-0.309 g/cm^3 [59].

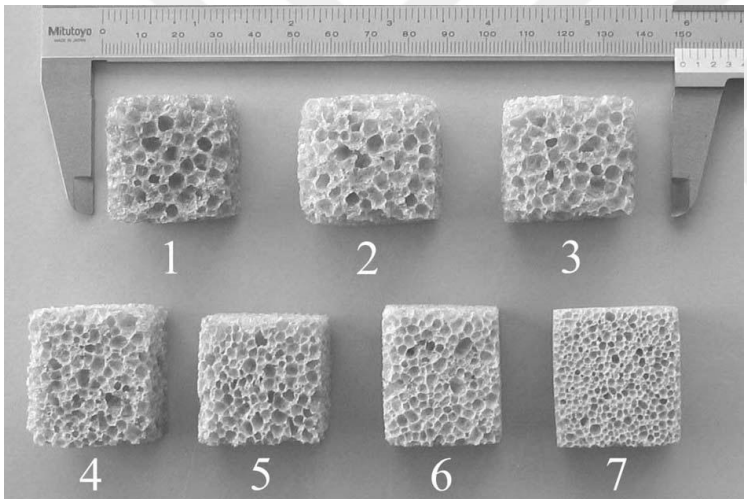


Figure 1.17. Foam glass samples produced by Tulyaganov et al. [59].

Brusatin et al. (2004) investigated a correlation between experimental conditions of glass and foaming agent (SiC) grain size, concentration, sintering temperature and time. According to the study, decreasing of glass powder particle size leads to smaller cell size and more homogeneous pore structure. When glass powder particle size is quite larger than foaming agent, agglomeration and separation inside the mixture can occur and inhomogeneous structure can be obtained. High sintering temperatures like 950-1000°C for 30 minutes holding time at peak temperature negatively affected pore structures and inhomogeneous cell size distribution were observed. When compressive strength and strut dimension were considered, it was seen that thicker struts provided

more dense foams and larger cells. It was explained that increasing of struts thickness resulted in different sized voids formation and decreasing of strut resistant. Increasing of foam glass density was achived with larger cell dimensions, on the other hand, smaller cells provided higher strength values on foam glass which has similar density. When glass grain size is larger, larger and inhomogeneous cell structure was observed thus density of foam glass also increases. However, high density is not a required property for insulation applications [68].

In the study conducted by Bayer (1980), borosilicate glass and SiC and Si_3N_4 foaming agent were investigated in foam glass production to elaborate their effects on pore size and pore structures of foam glass. In the experiments, fine powders of foaming agents (5 wt. % and <0.01 mm), CuO and kaolin minerals (<0.05 mm) were obtained as purest grade and mixed with borosilicate glass powder (<0.1 mm). Compositions were dry mixed with ball mill. Mixtures were pressed with using stainless steel moulds and cylindrical pellets with 15 mm diameter were obtained. Sintering process was applied by heating from room temperature to 950-1150ºC for 2 hours, holding at peak temperature for 5 minutes and very slow cooling with 0.5-2ºC/min. It was indicated that borosilicate glass have a tendency to crystallize to crystobalite when heated at foaming temperature (1000°C). Mechanical properties of partially crystallized foam glass are worse because of microcracks formation after cooling. To prevent crystallization, mineral components which react with softening glass were added and the glass viscosity increased by this way. Kaolin addition higher than 10 wt. % caused most effective results, while crystallization of crystobalite was seen in the foam glass without kaolin. As a result of the study, it was specified that preventing of crystobalite formation on glass can achieved with addition of 5 wt. % foaming agent and 30 wt. % kaolin at 1000-1100 ºC [60].

A study executed by Méar et al. (2007), mechanical, thermal and electrical behavior of foam glass were investigated by using SiC (<63 μm) and TiN (<10 μm) foaming agents with adding in a ratio of 4 wt. % and 5 wt. % into waste cathode ray tube (CRT) glass (<63 μm). CRT glass cullet was separated as funnel, panel and 2:1 mixture of funnel and panel glass mixture as feedstock to be used in the study. Later, arabic gum (2 mL of 1 wt.% aqueous solution) was added as binder, then the mixture was pressed at 5 tonnes by using uniaxial dry pressing and disc shaped samples with 40 mm diameter and 6 mm

thickness were obtained. Samples were sintered at 750ºC for 120 minutes under air atmosphere by using electrical furnace.

Expansion range in diameter of foam glass samples were measured as 10-100 %, while expansion in thickness ratios were in the range of 10-150 %. Microstructural characterization showed that using of SiC as reducing agent provided very fine cells with very narrow pore size distribution. On the other hand, using of TiN as reducing agent caused double distribution on the cells. Funnel glass addition leaded to higher porosity values as 84.5 % and 86.1 % with using SiC (5 wt. %) and TiN (4 wt. %), respectively. Required porosity values could not be obtained by using both SiC and TiN panel and mixed glass. Generally panel and mixed glass addition enhanced the compressive strength and failure stress of foam glass. On the other hand, increasing porosity in foam glass decreased mechanical properties like compressive strength and failure stress values. The lowest thermal conductivity and thermal diffusivity values were observed from the foam glass by using funnel glass and TiN as 0.08 $Wm^{-1}K^{-1}$ and $4.73x10^{-7}$ m^2s^{-1}, respectively. Depend on the high porosity, compressive strength was measured between 4-267 MPa and flexural strength was measured between 4-68 MPa. When dielectric properties was considered, it was seen that samples had lower conductivity than window glass. Finnaly it was also shown that obtained foam glass satisfied the required properties and could be used as insulating material [73].

Bernardo et al. (2007) conducted a study about glass foam production by using two different waste materials: recycled soda-lime glass and SiC-based wastes. The glass wastes were dry ball-milled and <37 μm sized powders were obtained. It was indicated that SiC-based polishing waste (<50 μm) contained about 50 wt. % SiC and some abraded (artistic) glass. The polishing waste was added into glass in the range of from 2.5 to 17.5 wt. %. Then, some selected samples were prepared by addition of MnO_2 powder (10 μm) in two different composition: (MnO/SiC-based waste: 1/5 or 1/3). Glass-SiC waste composition was mixed to homogenize for 30 minutes, then pressing was applied in a rectangular mould without using force. Sintering and foaming process were performed from the following shedule: Heating at 10°C/min rate to 950°C, holding at sintering-foaming temperature for 1 hour, expeditiously cooling from sintering-foaming temperature to 600°C with more than 10°C/min rate, then slowly cooling with nearly 1°C/min rate to 500°C and fast cooling to the room temperature with ≈5°C/min

rate. The purpose of fast cooling ("freezing") from sintering-foaming temperature to 600°C was performed to inhibit the pores disapperance in the microstructure which occur during the holding time. After then, slow cooling to 500°C was made at a temperature closed to the transformation temperature $T_g \approx 570$°C for annealing.

When obtained foam glass samples were examined, a significant decrease was obtained in relative density with increasing SiC-based waste into soda-lime glass. Addition of SiC-based waste with a ratio of 12.5 wt. % provided the minimum relative density with a value of $\phi=0.08$ and therefore the porosity content was measured as 92 % for this sample. Correspondingly, compressive strength values were decreased according to the relative density reduction. Addition of foaming agent caused a distinct decreasing on mechanical strength because of the cellular structure coarsening. Thus, high amount of foaming agent addition did not provide an advantage due to loss of mechanical strength. Also, it was noted that addition of SiC-based waste lower than 12.5 wt. % revealed very homogeneous microstructure. This homogeneous microstructures have high amounts of small pores with thin separation walls. It was indicated that different distribution of solid phase in the cell edges and faces provided positive effect on the mechanical strength.

Finally, the researchers concluded that soda-lime glass wastes could be transformed into glass foams with adding SiC-based polishing waste. The most proper result with high amount of foaming was achived from 12.5 wt. % SiC-based waste addition and at 950°C sintering temperature. It was shown that SiC addition ratio affected the mechanical strength which also depends on the homogeneity of foaming and microstructures [64].

Another study conducted by Méar et al. (2005), they attempted to change chemical environment of lead in the glass by adding of TiN or SiC reducing agents into lead silicate glass. The glass used in this study contains PbO (2-5 mol %) which was supplied as cathode ray tube industry waste. Glass powders were mixed with different amounts of reducing agents TiN or SiC (1, 3, 5, 7 or 9 wt.%). Mixtures were pressed by using uniaxial press and 40 mm diameter cylindrical shape samples were obtained. Sintering was applied at temperatures between 700-900°C; after about 90 minutes, glass foams were obtained with remarkable expansion. After SEM examination,

heterogeneous pore size distribution was observed for both TiN and SiC reducing agents (Figure 1.18).

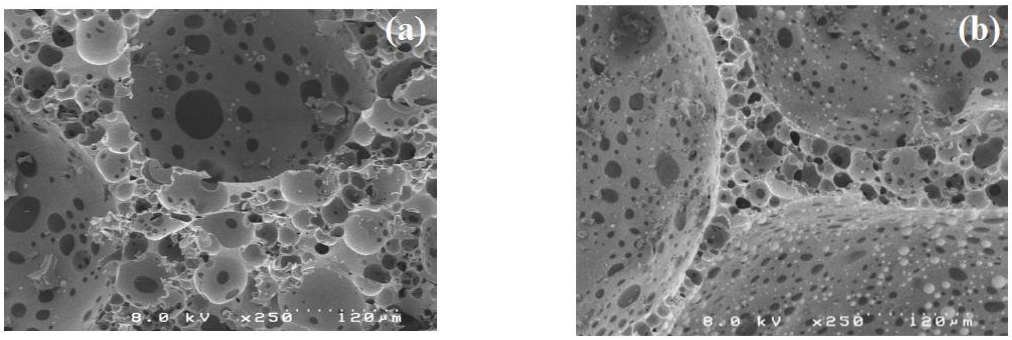


Figure 1.18. SEM micrograph of foam glass produced with **(a)** 5 wt. % SiC and **(b)** 5 wt. % TiN [62].

Foam glass samples were observed with irregular surface with a three dimensional lattice of voids and channels. Also, it was seen that small faceted droplets occurred on the glass surface. It was indicated that these droplets increased with increasing of reducing agent (SiC or TiN). EDS analysis showed that these droplets contained high concentration of Pb. At the end of this study, change in the chemical environment of the lead in the glass was observed by adding of reducing agents, TiN or SiC. Divalent cation Pb(II) contained in the vitreous network partially reduced to Pb(0). It was concluded that all lead oxide can be reduced nearly with addition of reducing agent (TiN or SiC) into lead silicate glass at proper sintering condition [62].

Hasheminia et al. (2012) investigated effect of PbO on foaming and crystallization behaviours of glass which based on diopside glass-ceramic composition with an addition of SiC as foaming agent. The main glass composition (indicated as F) was composed of 55.05 wt. % SiO_2, 13.61 wt. % Al_2O_3, 24.42 wt. % CaO, 6.92 wt. % MgO, 2.82 wt. % Na_2O and 3.02 wt. % K_2O. Different amount of PbO (5, 10 and 15 wt. %) was added into F glass and coded as F5, F10 and F15, respectively. Also, it was specified that the raw materials used in the study were reagent-grade chemicals with a purity of >99 %. Mixed glasses were melted in zircon crucibles at 1450°C for 2 h. Melted glass were quenched in cold distilled water and glass frits occurred. These glass frits were ground in electrical hard porcelain mortar for 2 h and glass powders were achieved with 4.5 µm mean particle size. SiC powder used in this study was mentioned as being commercial-grade and mean particle size was 12 µm. In the experimental

study, SiC powders were added with a ratio of 2 wt. % into ground glass frits and this composition was mixed in electric hard porcelain mortar for 15 min. The mixtures were press by using uniaxial press under 40 MPa; thus cylindirical pellets were obtained with 22 mm diameter and 6 mm thickness. After drying of these pellets at 100°C, sintering was applied with following schedule: Heating with a rate of 10°C/min, firing at temperature range of 850-1000°C and holding for 1 h.

According to DTA analysis, PbO addition caused a rising on crystallisation peak temperature while it was seen that peak intensity decreased with PbO increase. It was indicated that PbO addition supplied a reduction of crystallisation affinity of glasses. In addition, XRD results showed that diopside and gehlenite phases were precipitated at 1000°C in all glass. Temperature increasing caused a reduction of foam glass bulk density, the lowest value was obtained at between 900-1000°C.

Authors indicated that foaming ability in the lead containing glasses develops with decreasing of crystallisation peak intensities of diopside, thus affinity for crystallisation decreased. As a result of these properties, glass reacts with SiC particles and foaming occurs with gas evolving and foam glass products were achived with having low bulk density as 0.5 g/cm^3 [71].

In the study carried out by Méar et al. (2006), foam glass production was studied by using various Cathode Ray Tube (CRT) glasses, reducing and oxide agents. Screen and funnel glasses were evaluated separetely and as mixture in a ratio of screen:funnel:2:1. Glass compositions were mixed with reducing agents (SiC with various ratio of wt. 3.34, 5 and 6.68 % and TiN with various ratio of wt. 2.66, 4.00 and 5.32 %) and oxidising agent (MgO with various ratio of wt. 2, 3 and 4 %). Powder mixtures were granulated with using binder and pressed by uniaxial dry pressing. Sintering was applied at 800°C for 60 min.

Density, porosity and pore size values of foam glass samples were measured by Hg porosimeter. Bulk density values were varied from 0.51 to 0.99 g/cm^3, skeletal density values were calculated from 2.53 to 2.71 g/cm^3, open porosity values were measured from 63.6 to 81.0 %, closed porosity values were obtained changing from 0.9 to 2.7 % and total porosity values were measured from 65.1 up to 82.5 %. Both SiC and TiN reducing agents supplied high porosity around 80 %. Average pore size of the samples

which contain the agents with a ratio of 3.34 wt. % SiC + 2 wt. % MgO and 5 wt. % SiC + 3 wt. % MgO was found as 0.5 μm. In addition, pore size distribution of the sample produced with 5 wt. % SiC + 3 wt. % MgO was more narrow and homogeneous. Higher amounts of SiC caused heterogeneous pore size distribution and larger pore size diameter. Samples containing 6.68 wt. % SiC + 4 wt. % MgO had three different pore size distribution as 0.3, 3 and 100 μm. On the other hand, the foam glass samples produced with TiN reducing agent was found as having smaller pore diameter. It was indicated that the evolved gas after foaming reaction affected the pore size distribution. Authors reported that average pore size was measured smaller than the value measured by SEM micrograph observation. Because SEM analysis showed the real pore size of the cavities occurred after foaming process. It was explained that N_2 or CO_2 evolved gases (bubbles) caused interconnection between larger pores. According to the SEM micrographs, higher amount of reducing agent using (SiC or TiN) led larger pore size distribution [74].

In another research carried out by Méar et al. (2006), effects of temperature, reaction time and reducing agent content on foam glass microstructure were investigated. Lead containing cathode ray tube glass wastes were used as glass source, while SiC or TiN were added as reducing agent with various addition ratios. To see the effects of these parameters, reducing agents (SiC or TiN) were added with a ratio between 1 wt. % and 9 wt. %, then samples were sintered at various temperatures (750, 850 and 950°C) and holded at different reaction times between 30 and 90 min. When the effects of one parameter was investigated, the other two parameters were fixed to see the changing results on microstructures.

At the end of the study, it was indicated that increasing of mass percentage of SiC and reaction time caused an increase in pore size from 50 μm with homogeneous distribution to 100-300 μm with heterogeneous distribution. Increasing of reaction time caused an increasing on bimodal distribution with 1 mm average pore diameter. Similarly, temperature increase led to enlarging of pore diameter and it was pointed out that fine and homogeneous pore size distribution was achieved at 750°C. On the other hand, increasing of temperature to higher than 900°C caused negative effects on microstructure as a result of degredation and tearing of the pore walls were observed. Also, the pressure within the pores increased becuse of the higher temperature;

therefore, the number of interconnection windows and tearing of the pore walls increased, also.

It was concluded that increasing of parameters such as sintering temperature, SiC addition and reaction time resulted in more irregular pores and number of channels which connected the pores. The study showed that higher temperature resulted in higher pore size; accordingly, increasing the pore size had negative effect on the mechanical strength [65].

Francis et al. (2013) investigated density and water absorption capacity of glass-ceramic foams produced with SiC foaming agent. At the experimental study, granulated blast furnace slag and glass cullet (70 wt. %) were used as main raw material and glass source, while SiC (1-10 wt. %) was used as foaming agent. The effects of three different parameters; namely as sintering temperature (900, 950 and 1000°C), sintering time (30, 60 and 120 min) and foaming agent ratio (1, 5 and 10 wt. %) on the density and water absorption were investigated on the design study.

It was reported that the highest water absorption value was achieved at low sintering temperature, low reaction time and low addition ratio of foaming agent. XRD patterns of samples revealed two types of crystal phase: $CaSiO_3$ (wollastonite) and $Ca_2Mg_{0.75}Al_{0.25}Si_{1.75}O_7$. Also, unreacted SiC with a very small peak was observed at 900°C because of the incomplete oxidation reaction. Authors also indicated that wollastonite phase is usually desirable because it prevents polymorphic transformation during cooling from the foaming temperature to room temperature, and can be used for holding CO_2 and hinder the environmental pollution. Compressive strength values of samples were close each other and measured between 1.18 – 1.8 MPa. Optimum process parameters which provide the minimum density (0.61 g/cm^3) were determined as foaming at 998°C for 33 min with addition of 1.73 wt. % SiC foaming agent [75].

The key points of the literature survey are summarized in the APPENDIX 1-Table 1.

CHAPTER 2 - MATERIAL AND METHOD

2.1. Raw Materials

In the present study, sheet glass powder was designated as primary raw material. Within this scope, sheet glasses were obtained and it was transformed into glass powder by crushing and milling and considered as waste glass also. Secondly, to observe the effects of different purity graded foaming agents on the foam glass production process, different purity graded SiC powders (90, 95 and 98 % purity) were supplied for using as foaming agent. Also, carboxymethyl cellulose (CMC) was used for binding of glass and SiC powders. The raw materials used in the study and supplier companies were represented in Table 2.1.

Table 2.1. Raw materials used in the study.

Raw material name	Supplier company
Sheet glass	Şişecam Inc. Science and Technology Centre
90 % purity graded SiC	Süper Ateş Refractory Materials Industry Inc.
95 % purity graded SiC	Süper Ateş Refractory Materials Industry Inc.
98 % purity graded SiC	Süper Ateş Refractory Materials Industry Inc.
Binder (CMC)	ARIES

2.1.1. Glass powder

The sheet glass was the main raw material for glass foam production and it was supplied from Sisecam Science and Technology Centre (Cayirova, Kocaeli). At the beginning of the study, these sheet glasses were crushed into small particles by hand and jaw crusher. After then, wet and dry milling process were applied by using porcelain mills which have 170 mm and 200 mm inner diameter. Firstly, 2200 grams of crushed glass cullet particles were milled with adding 2,500 grams of water and 3,200 grams of alumina balls (at 16 mm, 18 mm, 21 mm, 22 mm, 28 mm ve 32 mm sized) at 72 rpm for 18 hours. After wet milling, glass samples were located in a crucible and dried at 110°C for 24 hours in drying oven. Dried glass powders were then dry milled with same alumina balls for 3 hours to prevent from agglomeration and fine glass powders were obtained finally. Glass powder preparing process stages were illustrated in Figure 2.1.

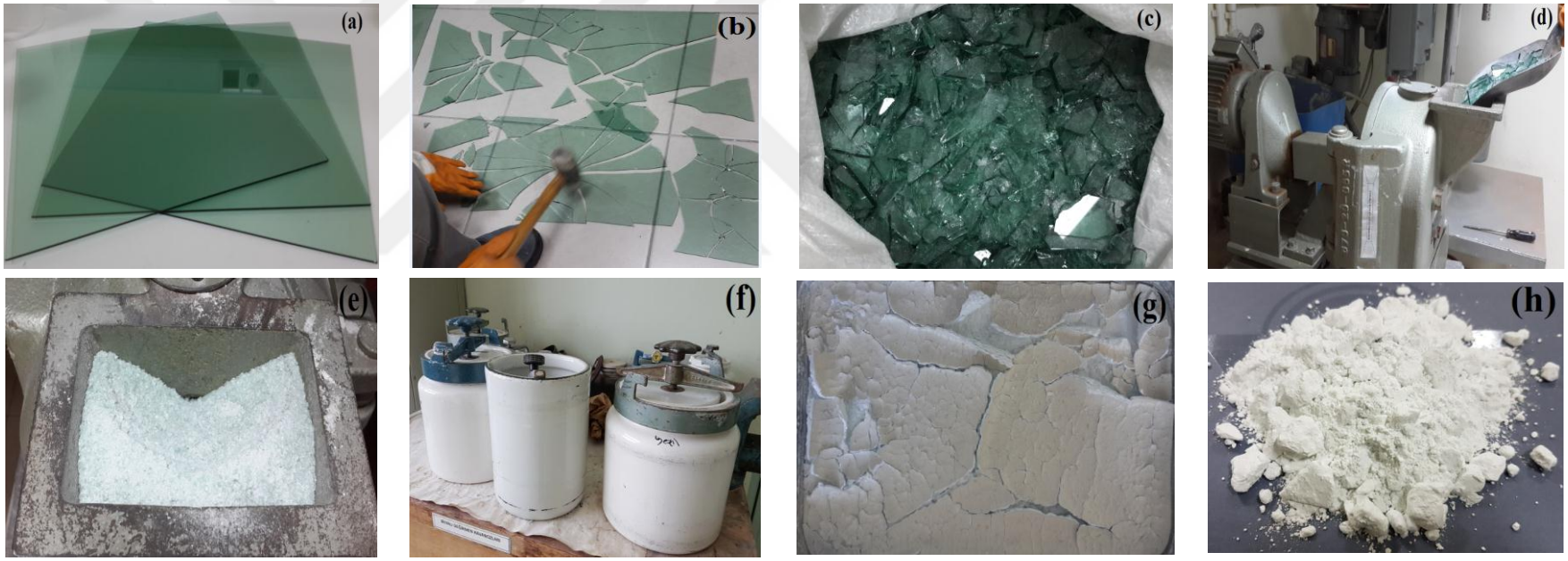


Figure 2.1. Waste sheet glass processing steps **(a)** sheet glass, **(b)** crushing step, **(c)** crushed glass cullet, **(d)** jaw crusher, **(e)** coarse crushed glass, **(f)** ball mills for wet milling, **(g)** dried glass after milling and **(h)** fine glass powder.

2.1.2. SiC powders with different purity grades

Foaming process was performed by using different purity graded SiC powders obtained from Süper Ateş Refractory Materials Industry Inc. (Ayazağa, İstanbul). As received SiC powder samples used in this study were illustrated in between Figure 2.2-2.4.



Figure 2.2. 90 % purity graded SiC powder.



Figure 2.3. 95 % purity graded SiC powder.



Figure 2.4. 98 % purity graded SiC powder.

2.1.3. Binder

Carboxymethyl cellulose (CMC) with a ratio of 0.02 wt. % was added to distilled water and mixed for 1 hour. This diluted CMC (Figure 2.5) was used as binder during the shaping of glass and SiC powder mixtures.



Figure 2.5. Diluted carboxymethyl cellulose binder.

2.2. Characterization Methods of Raw Material

Raw materials were characterized by particle size distribution, differential thermal analysis (DTA), thermal gravimetric analysis (TGA), semi-quantitative elemental analysis (XRF), mineralogical analysis (XRD) and scanning electron microscope (SEM) analysis.

2.2.1. Particle size distribution

Properties such as durability, chemical reactivity, opacity, fluidity and material strength of the materials depend on the grain size characteristics in the materials. The Mie Theory is used to measure the dimensions of small grains. The Mie Theorem takes into account both the breakage and permeability of the light around the grain size in the environment. In order to use the Mie model, it is necessary to know the fracture indices of both the sample and the environment [76]. Particle sizes of glass and SiC powders (90 %, 95 % and 98 % purity graded) were measured by using Malvern Mastersizer-2000 device (Figure 2.6). Measurement principle of this device is Mie scattering. Measuring range of Malvern Mastersizer-2000 is between 0.02 – 2000 μm particle size.



Figure 2.6. Malvern Mastersizer-2000 device.

2.2.2. Differential thermal analysis (DTA) - thermal gravimetric analysis (TGA)

Differential thermal analysis (DTA) and thermal gravimetric analysis (TGA) of raw materials were performed by SEIKO ExStar 6300 model DTA/TG thermal analysis device (Figure 2.7).

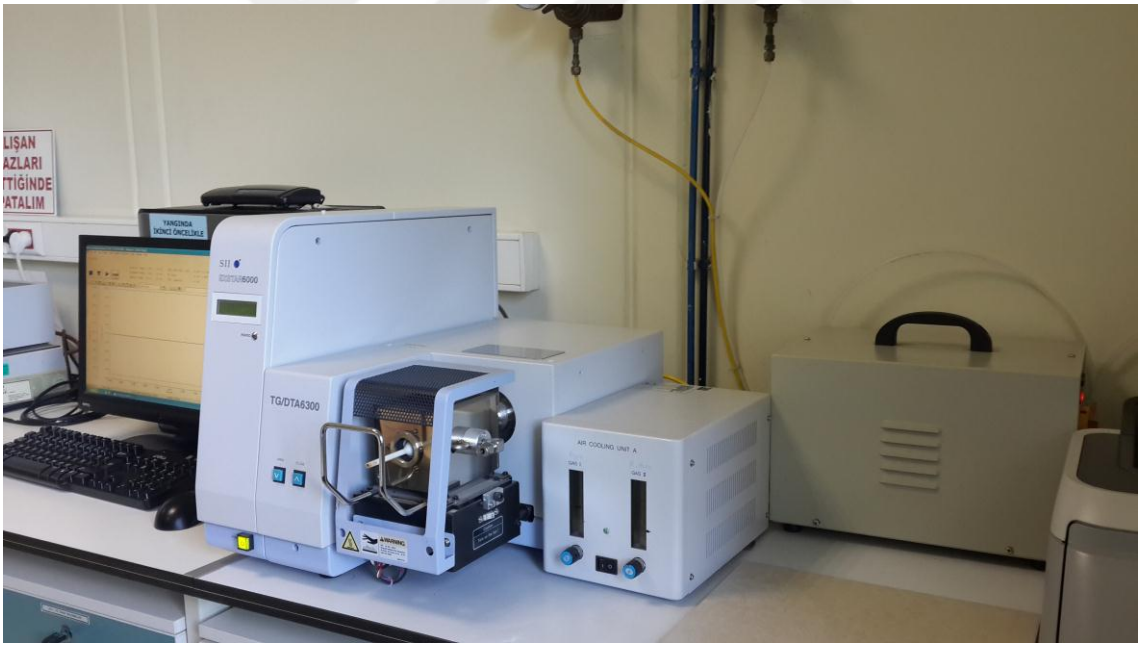


Figure 2.7. SEIKO ExStar 6300 model DTA/TG thermal analysis device.

DTA/TG thermal analysis device is used for definition of thermal changes, gravimetric changes and thermal behaviour within a material by applying heat. The temperature changes because of exothermic and endothermic reactions are determined by thermal analysis (DTA), while gravimetric changes (water loss, removing of organic matter) which occur within the material is measured by thermal gravimetric analysis (TGA).

With the help of DTA analysis, some properties like crystallographic transformations in minerals, melting, compositional degredation and water loss are determined. DTA/TGA analysis is used for graphically determine the weight and energy changes that minerals or different types of chemical substances show against the temperature increase. Especially, the weight loss of the ceramic raw materials due to the increase in temperature and the temperatures at which the reactions occurring in the structures are affected, the reaction type and the intensity, and thus the thermal behaviors of the raw materials are determined by this device.

2.2.3. X-Ray Fluorescence (XRF) – semi quantitative elemental analysis

Semi quantitative elemental analyses of raw materials were conducted by Philips PW-2404 model X-Ray Fluorescence (XRF) spectrometer device. XRF spectroscopy is one of the important methods used to determine the elemental composition of materials. According to the XRF device working principle, if the atom is excited by a high-energy radiation such as X-rays, this high energy input will bring the electrons in nearby orbits to higher energy levels. When excited electrons return to their initial energy levels, they return the gained excess energy in the form of X-rays with a wavelength of 0.1-50 Å. The emission of these secondary X-rays is called fluorescence emission. The wavelengths of these radiations given by the elements are different and distinctive for each element. In other words, these radiations are like fingerprints of that element. Quantity of the element is measured by determining the wavelength of the light, while concentration of the element is specified by measuring of the density of the detected radiation.

2.2.4. Mineralogical analysis (XRD)

Mineralogical analyses of raw materials were performed by SHIMADZU XRD-6000 Diffractometer device (Figure 2.8). Qualitative phases (mineralogical) and XRD patterns are measured by this device. According to theoretical working principle, X-rays with short wavelengths are sent over the sample to be tested. Light beams undergo diffraction from the three-dimensional crystal lattices of the material. Comparing the crystal structure of each mineral with different diffractions, it is revealed that the tested material which has mineralogical structure.



Figure 2.8. SHIMADZU XRD-6000 Diffractometer device.

2.2.5. Scanning electron microscope (SEM) and energy dispersive spectrometer (EDS)

Scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) analysis of 90, 95 and 98 % purity graded SiC raw materials were performed by scanning electron microscope "FEG-SEM JEOL JSM-6335F device". This device is used for microstructural characterization and semi quantitative micro chemical analysis of the materials.

2.3. Experimental Procedure

After raw material characterization, experimental study was designed based on the literature knowledge and to perform investigation of the effects of three different parameters which are illustrated in Table 2.2.

Table 2.2. Parameters and different variance researched in the experimental study.

Parameters	Variance
Purity grade of foaming agent (%)	90, 95 and 98
Composition ratio of foaming agent (wt. %)	1, 3, 5, 7 and 9
Sintering temperature (°C)	800, 825, 850 and 875

2.3.1. Flow chart

Experimental study steps were given in the general flow chart below (Figure 2.9).

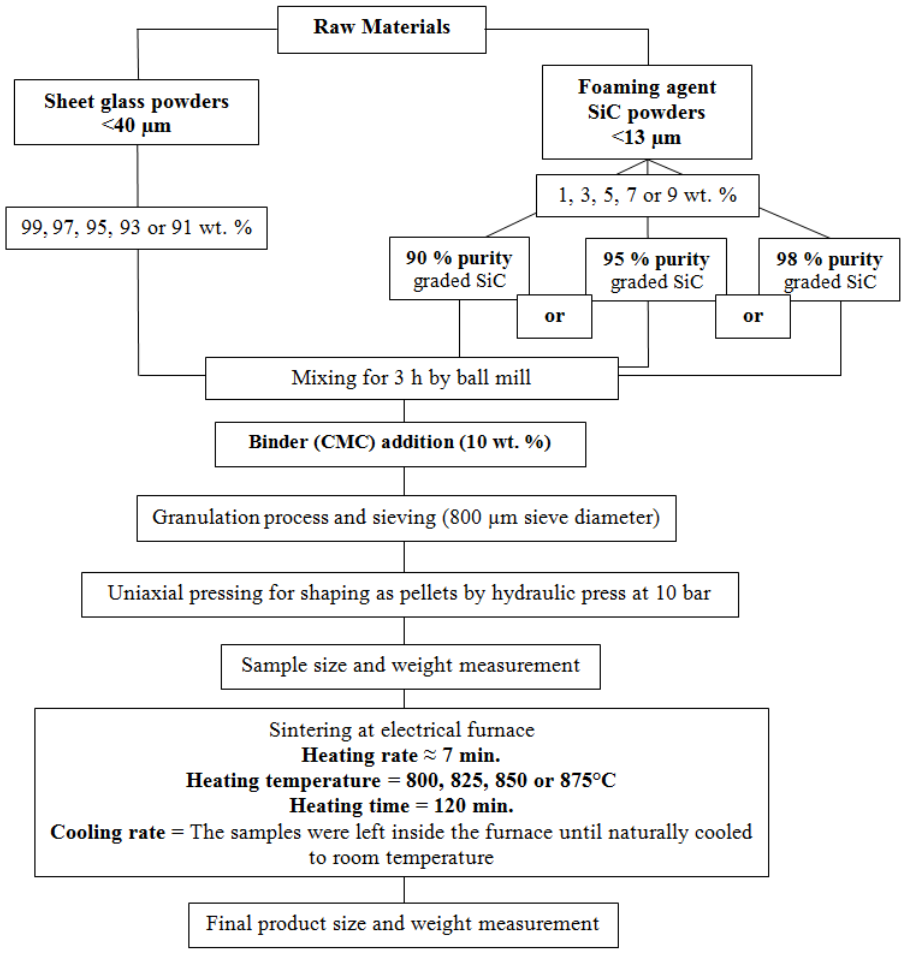


Figure 2.9. Flow chart of experimental study.

2.3.2. Sample preparation

Foam glass compositions were prepared by mixing of waste sheet glass powders and each purity graded SiC powders (90%, 95% or 98% purity graded) with different mixing ratios. Composition ratios were depicted in Table 2.3.

Table 2.3. Composition ratios of foam glass samples.

SiC composition ratio (wt.%)	Glass composition ratio (wt.%)
1	99
3	97
5	95
7	93
9	91

Prepared mixes were filled into rigid plastic container and mixed at ball milling device for 3 hours at 155 rpm (Figure 2.10). Nearly 10 alumina balls were used for 30 g of powder mixture to achieve more homogeneous mixture.

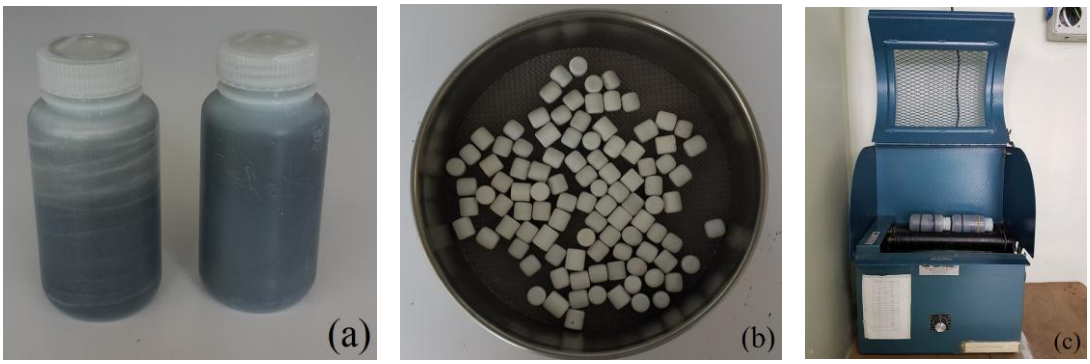


Figure 2.10. (a) Plastic containers, **(b)** alumina balls and **(c)** ball milling device.

2.3.3. Granulation and pressing procedure

Mixtures were granulated with addition of 10 wt. % CMC binder and sieved from -800 μm sieve to make ready for pressing. Figure 2.11 represents granulation process and an example for granulated sample.



Figure 2.11. Granulation process **(a)** addition of binder, **(b)** granulation, **(c)** sieving and **(d)** granulated sample.

After then, granulated samples were pressed under 10 bar (500 kg/cm^2) with using cylindrical heat-treated steel mould (23 mm Ø). Uniaxial hydraulic press used for the pressing studies was shown in Figure 2.12. Also, some pressed samples were illustrated in Figure 2.13.



Figure 2.12. Hydraulic press.

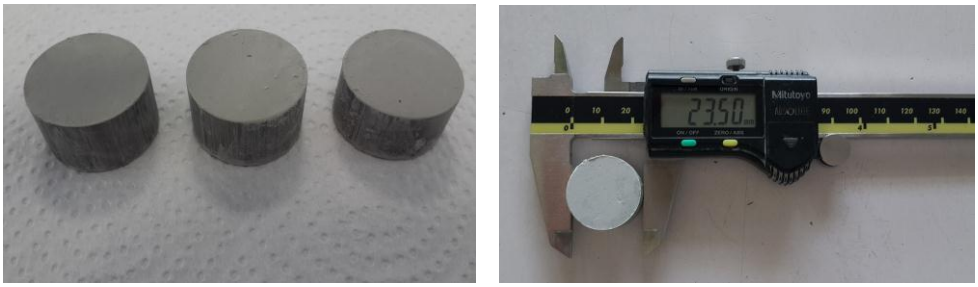


Figure 2.13. Some of the pressed samples.

2.3.4. Sintering process

After pressing process, foam glass samples were sintered at different temperatures with same heating rate and holding time by using PROTHERM PLF 120/45 model furnace (Figure 2.14). Sintering process was applied by following schedule: heating to sintering temperature (800, 825, 850 and 875°C) with 7°C/min heating rate, holding for 120 minutes at peak temperauture and natural cooling in the closed furnace down to room

temperature. Sintering schedules (Table 2.4) were coded as A, B, C and D for 800, 825, 850 and 875°C sintering temperatures, respectively.

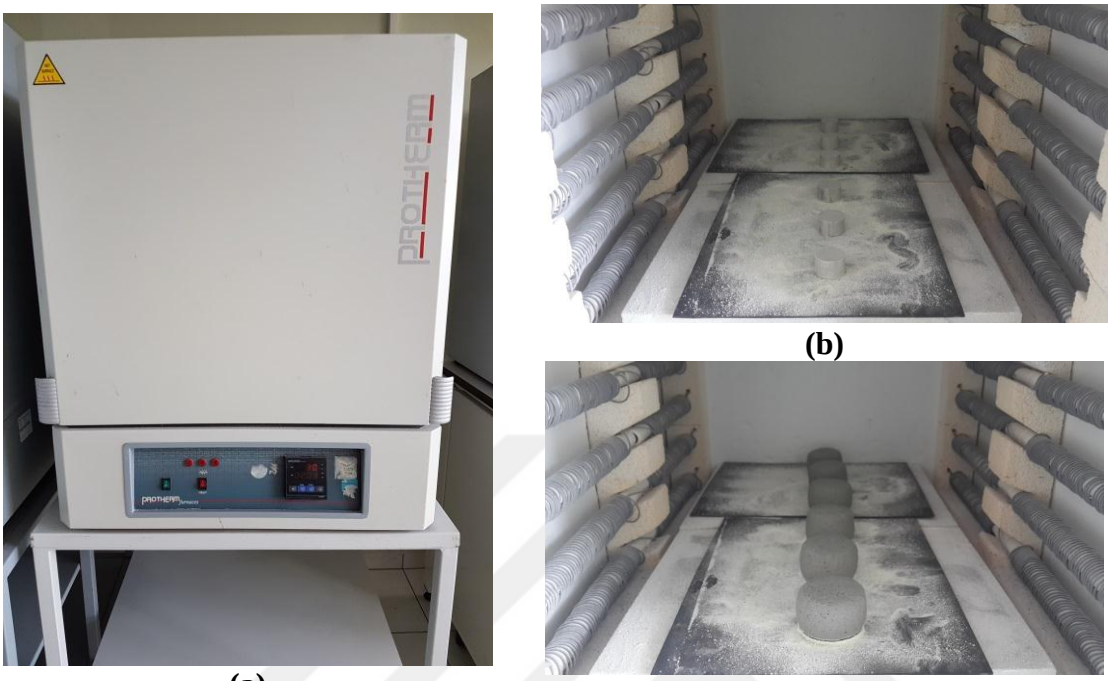


Figure 2.14. PROTHERM PLF 120/45 model furnace **(a)** outside view, **(b)** inside view before sintering and **(c)** inside view after sintering.

Table 2.4. Sintering schedules.

Sintering schedule code	Heating rate (°C/min)	Sintering temperature (°C)	Holding time at peak temperature (min)
A	7	800	120
B	7	825	120
C	7	850	120
D	7	875	120

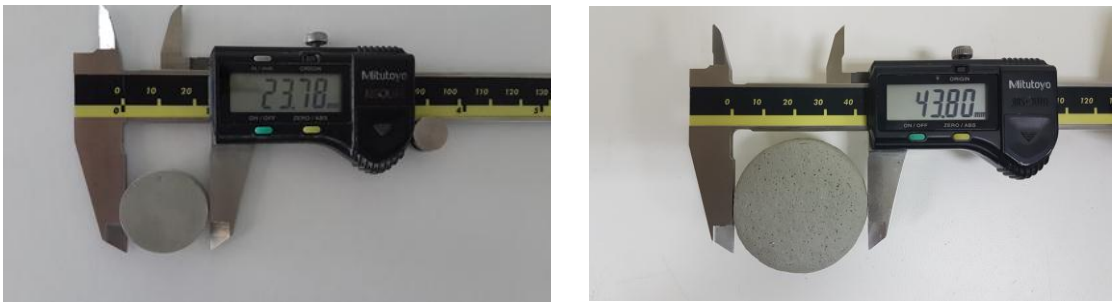
2.4. Characterization Methods of Foam Glass Samples

Foam glass samples were produced by using different purity graded foaming agents, different composition ratios and different sintering temperatures. After sintering samples were characterized to determine their physical, microstructural and chemical properties. Following tests and analyses were applied for sample characterization studies.

- Volume expansion ratio
- Mineralogical analysis
- Compressive strength
- Apparent density
- Short term water absorption by partial immersion
- Long term water absorption by total immersion
- Microstructural evaluation
- Thermal conductivity
- Total porosity
- Coefficient of thermal expansion
- Water vapor diffusivity strength

2.4.1. Volume expansion ratio

Volumetric changes of foam glass samples were calculated by measuring of diameter (mm) and thickness (mm) values of samples before and after sintering process (Fig.2.15). The formula used for measuring of volume expansion of the samples were given in Equation 2.1.



(a) **(b)**

Figure 2.15. Measuring of foam glass sample volume **(a)** before and **(b)** after sintering.

D_i = Initial dimension (before sintering), (mm)

H_i = Initial height (mm)

D_f = Final dimension (after sintering), (mm)

H_f = Final height (mm)

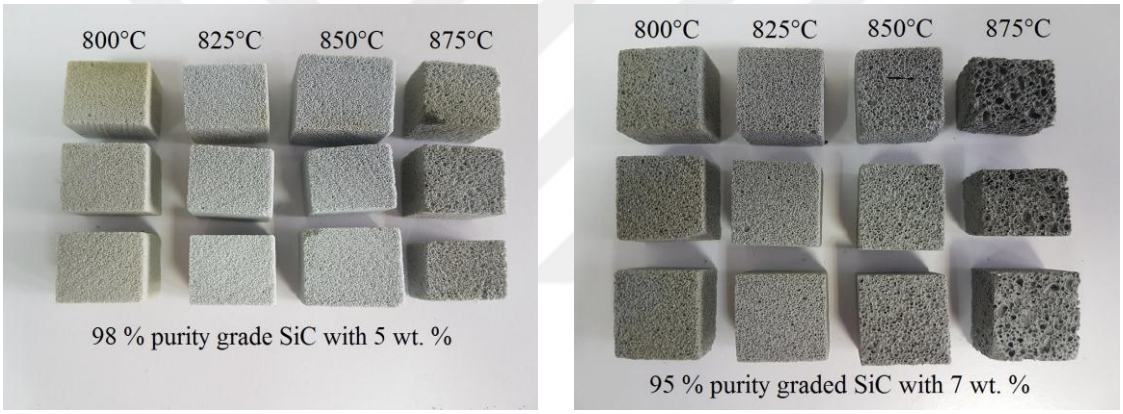
$$\text{Volume expansion ratio (\%)} = [[\pi(D_f/2)^2 x h_f]-[\pi(D_i/2)^2 x\ h_i]]/[\pi(D_i/2)^2 x h_i]x100 \qquad (2.1)$$

2.4.2. Mineralogical analysis (XRD)

Mineralogical analyses and XRD patterns of foam glass samples were also measured by SHIMADZU XRD-6000 Diffractometer device. To specify the effect of foaming agent purity grades, composition ratios and sintering temperatures, obtained patterns were overlaid according to the investigated parameter.

2.4.3. Compressive strength

Three cubic samples were prepared by cutting for each foam glass group (Figure 2.16) for compressive strength tests. Then, three measurements were performed for each sample. Compressive strength tests of foam glass samples were performed by Zwick Z250 Universal test device (Figure 2.17).



(a) **(b)**

Figure 2.16. Some foam glass samples prepared for mechanical tests **(a)** 98 % purity graded SiC with a ratio of 5 wt. %, **(b)** 95 % purity graded SiC with a ratio of 7 wt. % sintering at 800, 825, 850 and 875°C, respectively.

The compressive strength is usually calculated with using the Equation 2.2.

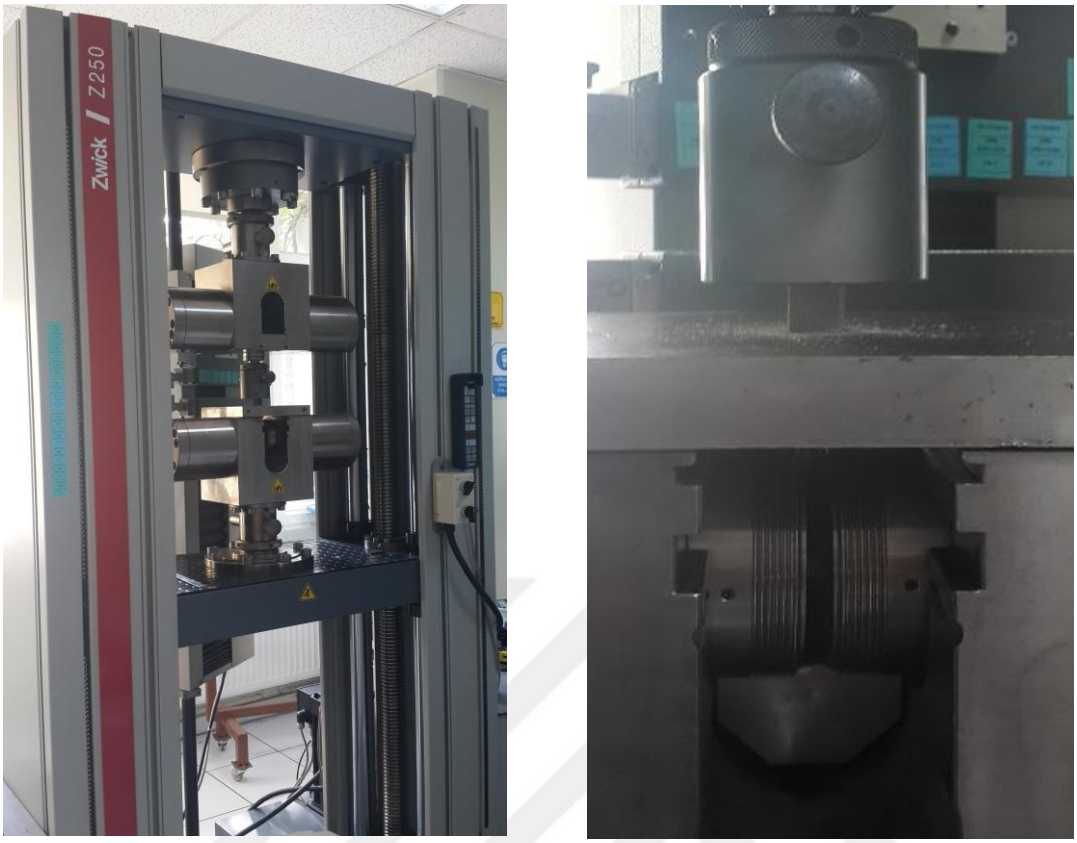
$$б = F_{max} / A \quad (2.2)$$

б : Compressive strength

F_{max} : Maximum force (or load)

A : The area perpendicular to the F_{max}

In this study, maximum force was determined in the MPa unit by using the compressive test device. Because of the other insulation materials and commercial foam glass materials present the compressive strength in the unit “MPa”, the maximum loads in MPa were considered.



(a) **(b)**

Figure 2.17. (a) Zwick Z250SN5A device and **(b)** an example during compressive test.

2.4.4. Apparent density

Density of the foam glass samples were measured by using the standard TS EN 1602:2013-06 "Thermal insulating products for building applications-Determination of apparent density" [43]. According to the TS EN 1602 standard, volumes of the test specimens were calculated by measuring the length, width, and thickness. Then, each test specimens were weighted. Finally, the apparent overall density (ρ_a, in g/cm^3) was calculated by the Equation 2.3:

$$\rho = m/V \ (g/cm^3) \qquad (2.3)$$

Where

m: Mass of the test specimen (g)

V: Volume of the test specimen (cm^3)

2.4.5. Short term water absorption by partial immersion

Foam glass samples were measured according to the TS EN 1609:2013-06 “Thermal insulating products for building applications-Determination of short term water absorption by partial immersion” standard [44]. This test method is applied for observing the water absorption behavior of the foam glass samples within 24 h. TS EN 1609 offers two method (method A or method B) for measuring the water absorption by partial immersion. In this study, Method A was chosen for short term water absorption by partial immersion test.

Method A (drainage):

Initial mass (m_0) of samples were weighted, then these samples were located in the empty water tank. Later, water was added until the bottom half of the sample is below the surface of the water (Figure 2.18). Water level was controlled to be constant during the test. Test samples were removed from the water tank after 24 h; then they were drained for (10 ± 0.5) min by locating on a perforated stainless steel (mesh) and holding at 45° (Figure 2.19). Finally, samples were weighted to specify their mass as m_{24}.

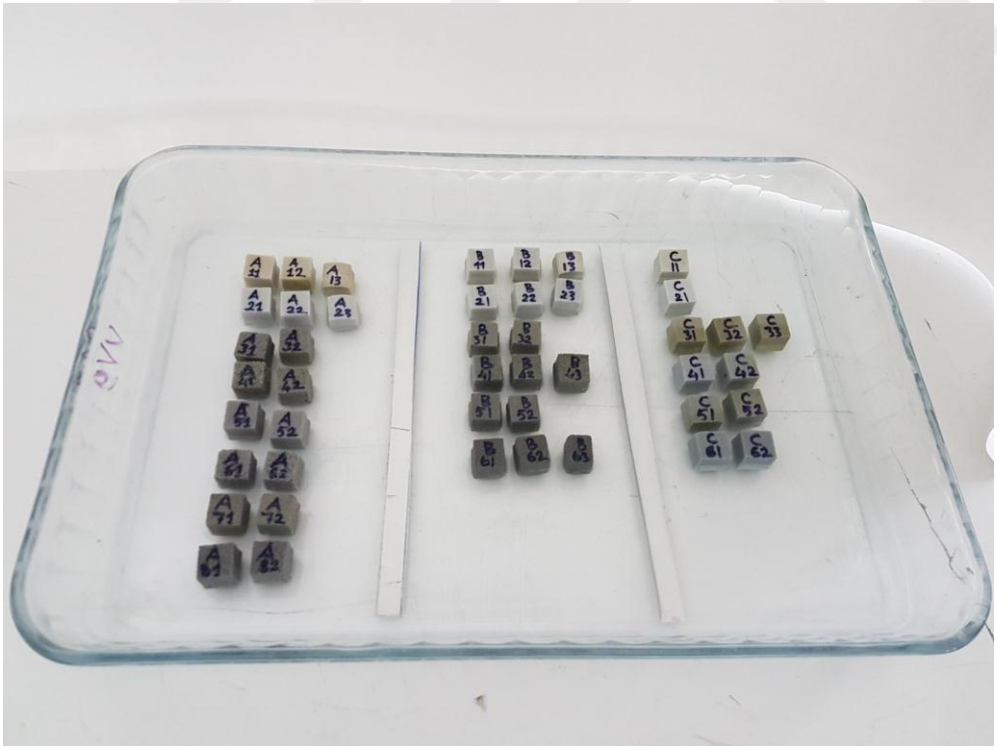


Figure 2.18. Illustration of short term partial immersion test.

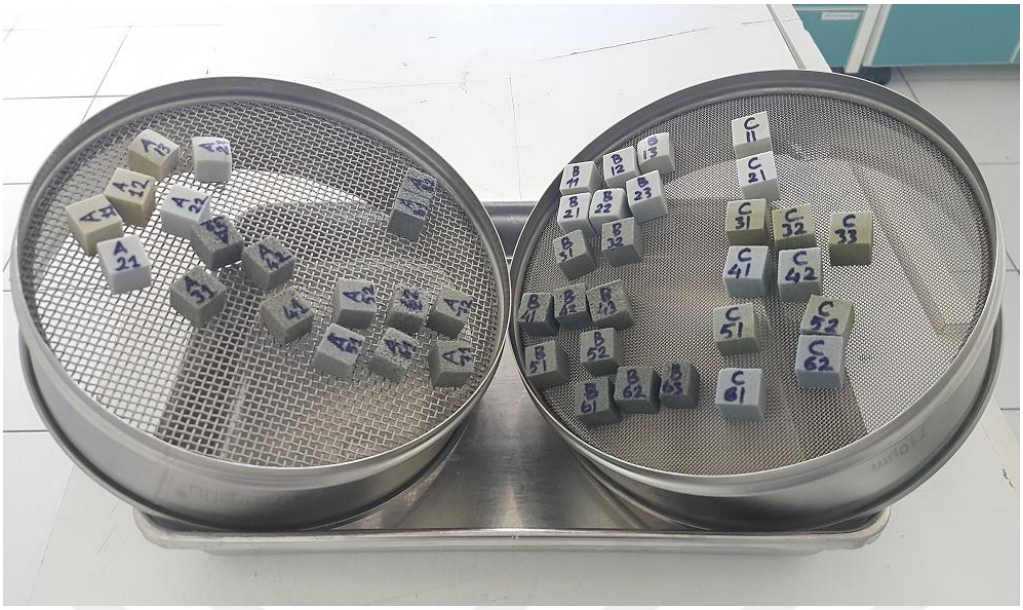


Figure 2.19. Illustration of drainage process.

Short-term water absorption by partial immersion (W_p) values were calculated in kg/m^2 with using the Equation 2.4:

$$W_p = (m_{24} - m_0)/A_p \quad (2.4)$$

Where

m_0 : Initial mass of the test sample (kg)

m_{24} : Mass of the test sample after partial immersion for 24 h (kg)

A_p : Bottom surface area of the test specimen (m^2)

2.4.6. Long term water absorption by total immersion

Long term water absorption by immersion were measured on foam glass samples according to the TS EN 12087:2013-06 "Thermal insulating products for building applications-Determination of long term water absorption by immersion" standard [45]. The long term water absorption by total immersion is determined by measuring the change in mass of the test specimen, totally immersed in water, over a period of 28 days. TS EN 12087 offers two methods (method A or method B) for measuring the water absorption by total immersion. In this study, Method 2A (drainage) was chosen for long term water absorption by total immersion test.

Method 2A:

Initial mass (m_0) of samples were weighted and dimensions of samples were measured to determine initial volume (V). Then, these samples were placed in the empty water tank and located a sufficient load to hold the test specimen totally immersed in water. Later, distilled water was added to the tank until the top faces of the samples were below the surface of water. Water level was controlled to be constant during the test. Test samples were removed from the water tank after 28 days; then, they were drained for (10 ±0.5) min by locating on perforated stainless steel (mesh) and holding at 45°. Finally, samples were weighted again to measure their mass, m_{28}.

Long term water absorption by total immersion, W_{lt}, was calculated in volume percent (%) by using the formula (Equation 2.5) below:

$$W_{lt} = [(m_{28}-m_0)/V] \times (100/\rho_w) \quad (2.5)$$

m_0 : initial mass of test specimen (kg)

m_{28} : mass of the test specimen after total immersion for 28 days in method 2A (kg)

V : initial volume of the test specimen (m^3)

ρ_w : density of water, assumed to be 1000 kg/m^3

2.4.7. Microstructural evaluation (SEM)

Microstructural characterization of foam glass samples were also conducted by FEG-SEM JEOL JSM-6335 F device (Scanning Electron Microscope). A voltage of 10 kV or 15 kV was applied in all observations and magnification of x30 was used for foam glass samples. This device is used for characterization of material's microstructures and analyzing for semi-quantitative micro analytic methods. To examine the microstructural differences of foam glass, some samples were selected according to the variation of foaming agent purity grade, sintering temperature and foaming agent addition ratio.

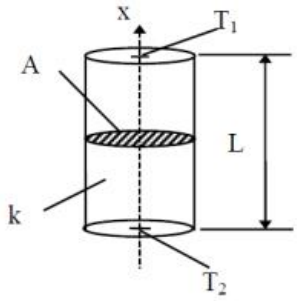
2.4.8. Thermal conductivity

Heat conduction term is used for heat transition from the hot zone to the colder zone within a solid material or stationary fluid. If there is a temperature difference within a solid body, the heat transforms from the high temperature region to the low temperature

region by conduction. The conduction heat transfer is determined by Fourier's law based on experimental observations. According to the Fourier's law, the amount of heat transfer in any direction (for example in the x direction) is proportional to the temperature gradient in the x direction (the amount of temperature change) dT/dx and the area (A) perpendicular to the heat transfer direction [77]. The mathematical expression of Fourier's law was indicated in the Equation 2.6 [78].

$$Q = -kA(dT/dx) \quad (2.6)$$

Q represents the amount of heat passing in the x direction and over this diagonal area in the x direction. The rate constant k is called the heat transfer coefficient and is a property of the material. The unit "k" is also used with the symbol "λ", and its unit in the SI unit system is W/mK. Negative symbol (-) in the equation 1.4 refers the direction of heat transfer. According to the Fourier law, calculation method of heat transfer within the homogeneous and isotropic cylinder which has different surface temperature (T1>T2) was illustrated in Figure 2.20 [77].



$$\dot{Q}_x = -kA\frac{\Delta T}{\Delta x} = kA\frac{T_1 - T_2}{L}$$

Figure 2.20. Illustration of thermal conductivity within a cylindrical material [77].

The thermal conductivity values of foam glass samples were also measured according to the Fourier law. Thermal conductivity analysis of foam glass samples were performed by using C-Therm Technologies TH89-05-00400 model thermal conductivity device by Ceramic Research Centre in Eskişehir. Samples to be measured are prepared with minimum 25 mm diameter and minimum 2 mm thickness. Measuring range of the thermal conductivity device used in this study is specified as 0 - 10 W/mK.

2.4.9. Total porosity

Porosity of foam glass samples were measured by applying mercury porosimetry method with using Quantachrome Corporation, Poremaster 60 device in the Middle East

Technical University Central Laboratory. Mercury porosimeters operate according to the physical principle that an inert and non-wetting fluid cannot enter small pores unless adequate pressure is applied. The relationship between the applied pressure and the pore diameter is obtained by the Washburn Equation 2.7:

$$D= (-4\gamma\cos\theta)/P \quad (2.7)$$

P : Applied pressure

D : Diameter of pore

γ : Surface tension of mercury (480 dyne/cm)

θ : Contact angle between mercury and pore wall (usually 140°)

To generate pore size / volume distributions, mercury intrusion data is used. The porosimeter device has two sample chamber, including low pressure (up to 50 psi) and high pressure (up to 55,000 psi). If the diameter of pores is between 200-4 μm, low pressure is applied; otherwise, if the diameter of pores is between 10.66-0.004 μm, then high pressure is applied to determine the pore size distribution. Because of the diameter of the pores of foam glass specimen produced in this study is in the range of 200-4 μm, low pressure was applied to measure total porosity (%) and pore size distribution [79].

2.4.10. Coefficient of thermal expansion

NETZSCH DIL 402 C/3/F model dilatometer was used to measure coefficient of thermal expansion (K^{-1}) values of selected foam glass samples. The change in the dimension of the sample during heating is measured by this method. According to the working principle of dilatometer device, standard polycrystalline Al_2O_3 rod is used as reference. During heating, changing of sample dimension is transmitted to the inductive transducer with Al_2O_3 rod. Electric signals occurring from continuous dimensional change in the transducer are transmitted to the control cabin, and then dimensional change is measured and saved.

Foam glass test samples were cut in the cylindrical shape with 8 mm Ø diameter and 25 mm long. Then, coefficients of thermal expansion (K^{-1}) values were measured between 28-300°C. Figure 2.21 shows an example for NETZSCH DIL 402 C/3/F model dilatometer device.



Figure 2.21. NETZSCH DIL 402 C/3/F model dilatometer device.

2.4.11. Water vapor diffusivity strength

Water vapor diffusivity strength (μ) value of selected foam glass sample was measured by Izmir Katip Çelebi University, Engineering and Architecture Faculty, Civil Engineering Department, Department of Construction Materials, Construction Materials R&D Laboratory according to the standard TS EN ISO 12572:2001-11 [48]. "Hygrothermal performance of building materials and products - Determination of water vapour transmission properties" was used to determine water vapor diffusivity strength (μ) value. Also, water vapor diffusivity (δ) and water vapor flow intensity (g/m^2h) values were measured by this method.

CHAPTER 3 - RESULTS & DISCUSSION

At the beginning of the study, raw materials were characterized by using several techniques including particle size distribution, differential thermal analysis (DTA) - thermal gravimetric analysis (TGA), X-Ray Fluorescence (XRF) - semi quantitative elemental analysis, mineralogical analysis (XRD), scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) investigations. Then, experimental procedure was planned and foam glass samples were produced by applying different parameters like sintering temperature, foaming agent purity grade and addition ratio. Finally, fired samples were characterized to determine following properties: volume expansion ratio, mineralogical phases, compressive strength, apparent density, short term water absorption by partial immersion, long term water absorption by total immersion, microstructural examination, coefficient of thermal conductivity, total porosity, coefficient of thermal expansion and water vapor diffusion resistance.

This chapter will be represented the raw material and fired foam glass sample characterization results in detail. Results were presented, discussed and compared with desired properties; also the relations between the characteristic properties were also investigated.

3.1. Characterization Results and Discussion of Raw Materials

3.1.1. Particle size distribution analysis

Initial particle size of raw material is important parameter for foaming process. Based on the literature knowledge, fine ground glass powders is more suitable to achieve more homogeneous and small cells. Also, foaming agent and glass particle size distribution should not be much different, otherwise, cavity structure and inhomogeneous porosity will dominate the foam glass. Within this view point, glass cullet was crushed and milled to have fine powder and achieved d_{90}: <40.02 μm sized powders. 90 and 98 % purity graded SiC powders were supplied as having very fine grain sizes (d_{90}: <13.5). However, 95 % purity graded SiC powder could achieve with larger grain size (d_{90}: 109.735 μm), so it was milled for 4 h by jet mill and obtained fine ground powders (d_{90}:< 7.5 μm) was used at the experiments. Table 3.1 shows the particle size results of

glass and SiC powders which are used in the study. Particle size distribution results are also given in Figure 3.1-3.14.

Table 3.1. Particle size results of glass and SiC raw materials.

Particle Size	**Waste sheet Glass Powder**	**SiC** ***(90 % purity)***	**SiC** ***(95 % purity)***	**SiC** ***(98 % purity)***
d_{10} (µm)	2.77	1.12	0.14	4.28
d_{50} (µm)	14.69	4.57	0.20	7.71
d_{90} (µm)	40.02	13.49	7.43	13.45

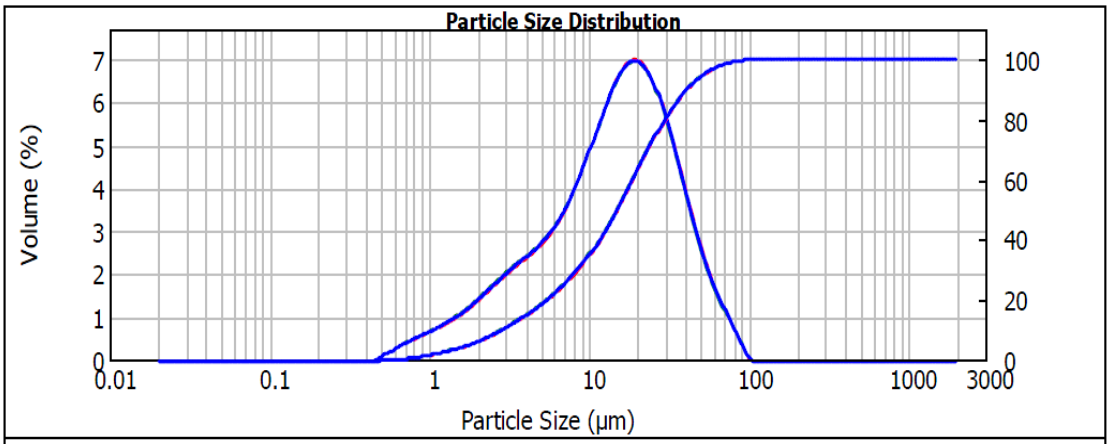


Figure 3.1. Particle size distribution of waste sheet glass powders.

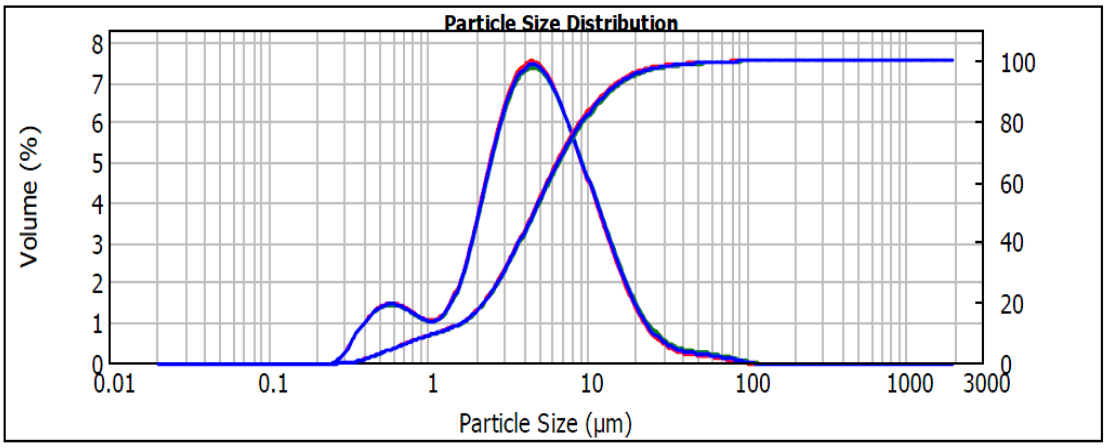


Figure 3.2. Particle size distribution of 90 % purity graded SiC powders.

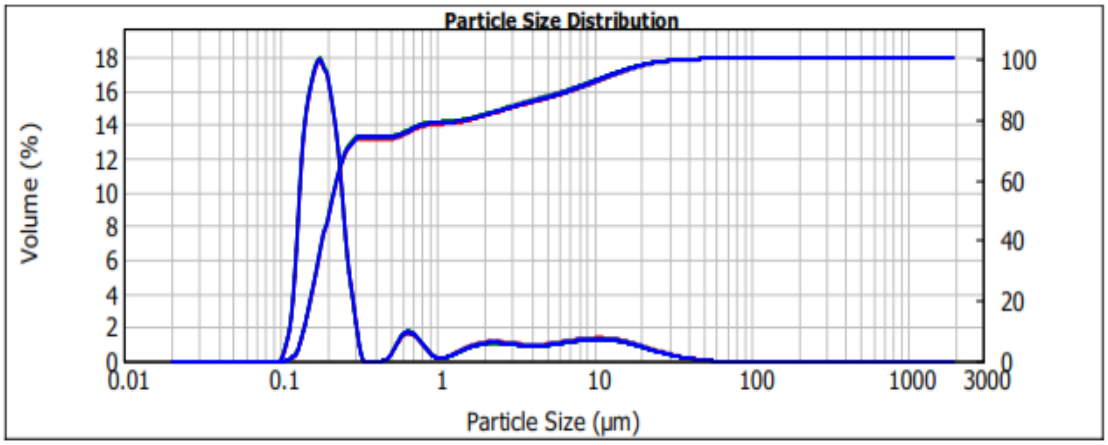


Figure 3.3. Particle size distribution of 95 % purity graded SiC powders.

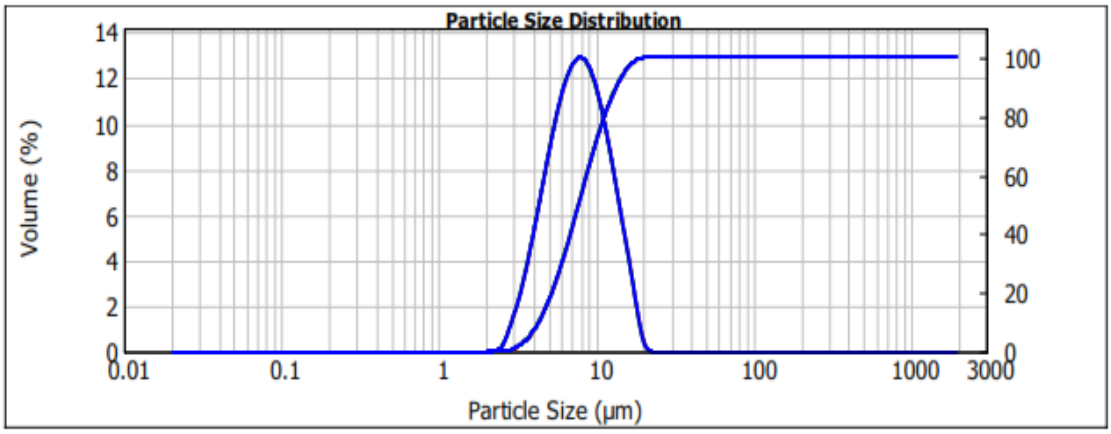


Figure 3.4. Particle size distribution of 98 % purity graded SiC powders.

3.1.2. Differential thermal analysis (DTA) - Thermal gravimetric analysis (TGA)

DTA/TG analysis provides insight about the thermal behavior of raw materials and sintering behaviour in particular. DTA/TG results of raw materials were given in Figure 3.5 – Figure 3.8. From the glass powder curves, it was seen that glass transition temperature (T_g), crystallization temperature (T_c) and softening point (T_s) were 580, 708 and 750°C, respectively. These findings lead to determine foaming temperature. By taking into consideration of literature knowledge, it was decided that sintering of foam glass samples would be above 750°C. In addition, different purity graded foaming agents gave different weight loss at 33-1000°C. Weight loss decreased with increasing purity grade of SiC powders. This is because of the fact that high purity SiC has higher melting point, thus there was no weight loss on 98 % purity graded SiC, while the highest weight loss occurred at 90 % purity graded SiC powder. The whole information

gives us a clue about foaming behavior. It was estimated that the foam glass samples with using 90 % purity graded SiC powder can be achieved at lower foaming temperature, on the other hand 98 % purity graded SiC foam glass will need higher temperature to observe required porous structure.

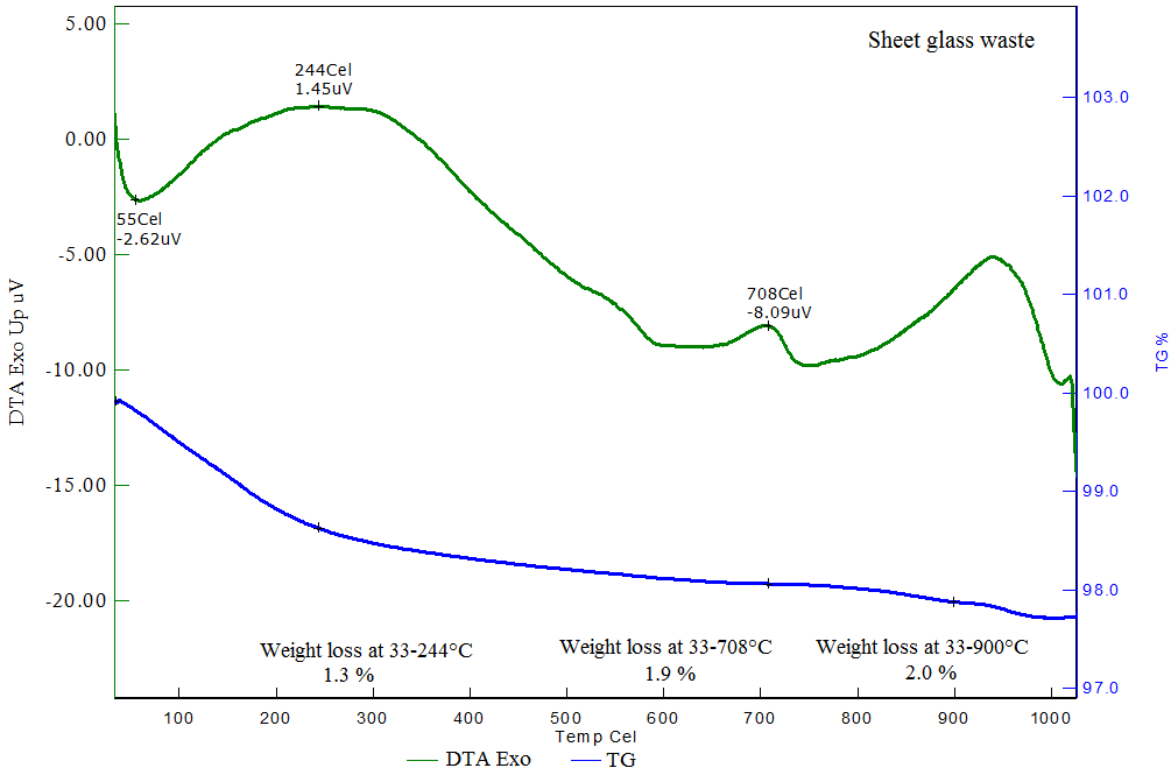


Figure 3.5. DTA/TG Analysis result of waste sheet glass.

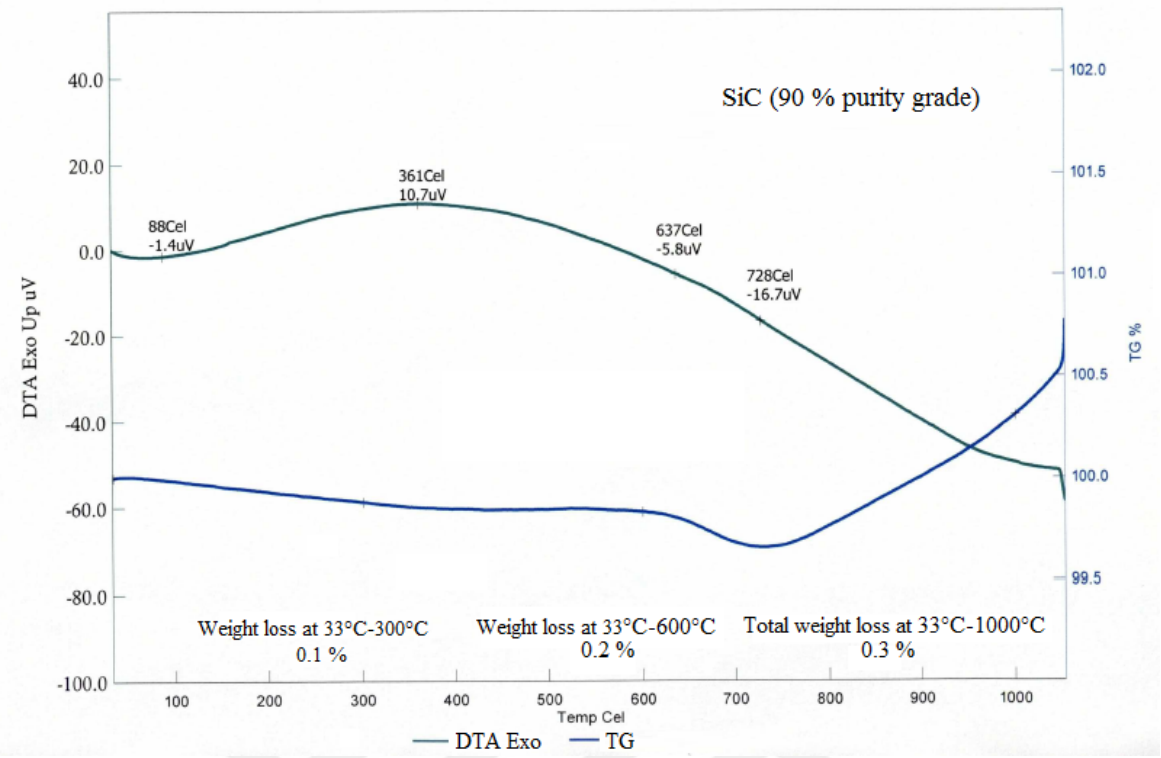


Figure 3.6. DTA/TG Analysis result of 90 % purity grade SiC.

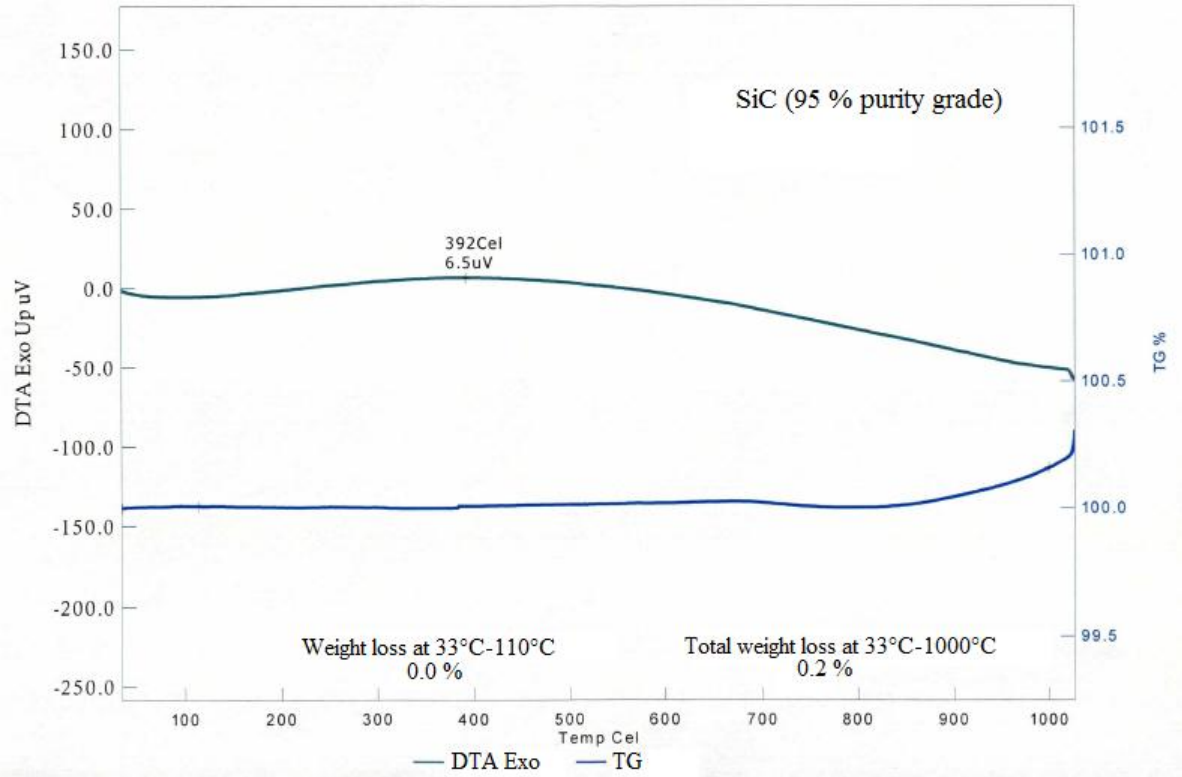


Figure 3.7. DTA/TG Analysis result of 95 % purity grade SiC.

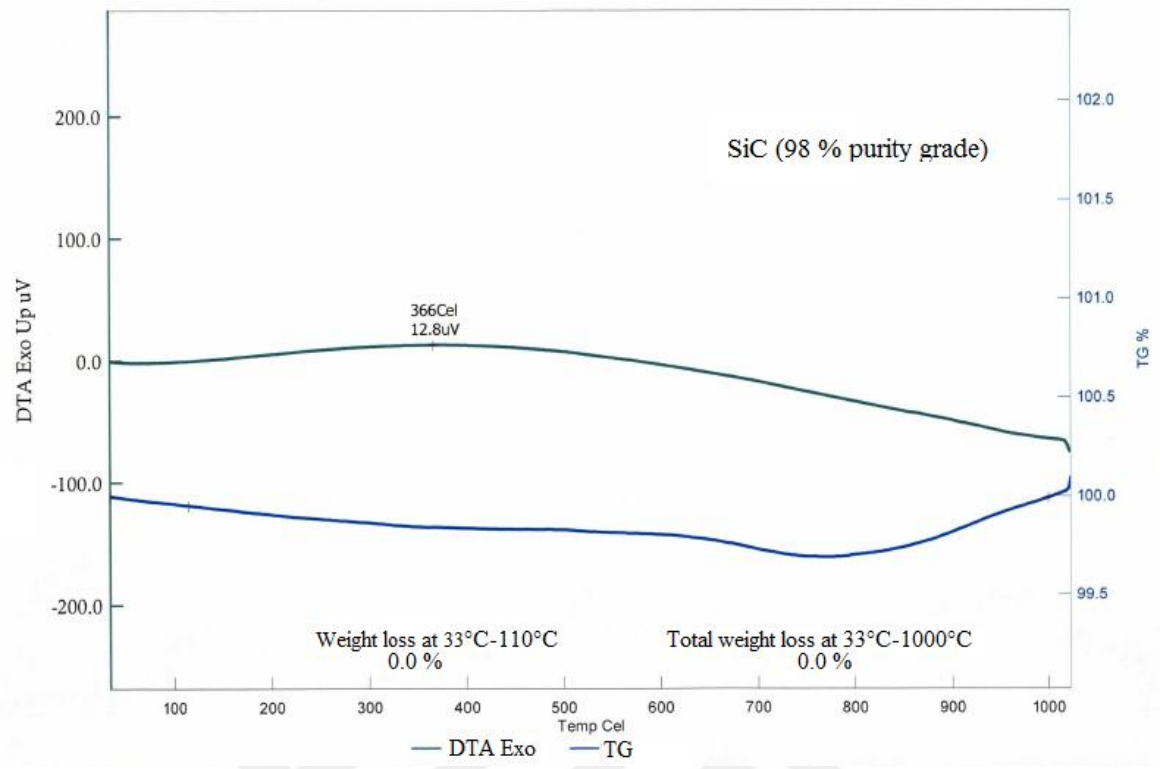


Figure 3.8. DTA/TG Analysis result of 98 % purity grade SiC.

3.1.3. X-Ray Fluorescence (XRF) - semiquantitative elemental analysis

Semiquantitative elemental analysis results of glass powder and 90 % purity graded SiC were shown in Table 3.2, also oxide contents of waste sheet glass powder were given in Table 3.3. Because of its influence on the thermal behavior, oxide content of glass is quite important. Na_2O and K_2O decreases the melting point of glass and makes easier the softening. CaO and MgO contents are also important for enhancing the strength. In addition, Al_2O_3 stabilizes the viscosity of glass at softening temperature, thus glass powders can hold the evolved gaseous species without spreading out from the body. Moreover, it was thought that the iron content of sheet glass causes the green colour.

Table 3.2. Semiquantitative elemental analysis results of waste sheet glass and SiC powders.

Element	**Waste Sheet Glass (%)**	**SiC *(90 % purity)* (%)**
Al	0.784	0.074
C	-	29.778
Ca	7.211	<<
Cl	0.304	-
Cr	<<	<<
Fe	0.971	0.201
K	0.057	-
Mg	3.900	-
Mn	<<	-
Na	6.550	-
Ni	-	0.021
O	46.912	0.218
P	0.242	-
S	0.278	<<
Si	32.737	69.630
Sr	<<	-
Ti	0.044	<<
V	-	0.073
Zn	<<	-
Zr	0.010	0.006

Table 3.3. Oxide contents of waste sheet glass powders.

Oxide	**Waste sheet glass powder (%)**
SiO_2	70.036
CaO	10.090
Na_2O	8.830
MgO	6.467
Al_2O_3	1.481
Fe_2O_3	1.388
SO_3	0.694
P_2O_5	0.555
Cl^-	0.304
TiO_2	0.074
K_2O	0.069
ZrO_2	0.013

3.1.4. Mineralogical analysis (XRD) results of raw materials

Mineralogical phases and chemical formulas of raw materials were given in Table 3.4 with JCPDS (Joint Committee on Powder Diffraction Standards) numbers. Raw materials XRD patterns were illustrated in Figure 3.9-3.12.

Table 3.4. Mineralogical analysis (XRD) results of waste sheet glass and SiC powders.

JCPDS #	**Mineralogical phase**	**SiC** ***(90 % purity)***	**SiC** ***(95 % purity)***	**SiC** ***(98 % purity)***	**Waste sheet glass**
42-1360 29-1127	Moissanite, **SiC**	* *	* *		*Amorphous*
4-2-9068 4-8-1656	Silicon Carbide, **SiC**			* *	
27-1402	Silicon, **Si**	*			
26-1076	Carbon, **C**			*	

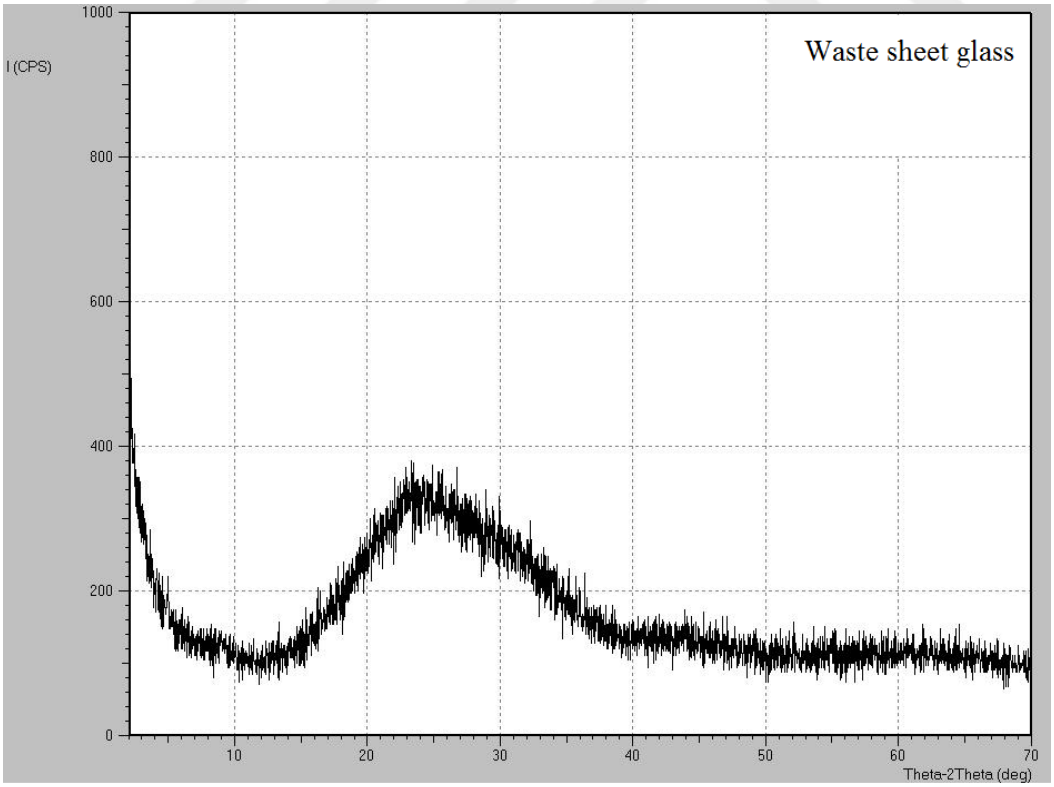


Figure 3.9. XRD pattern of waste sheet glass powders.

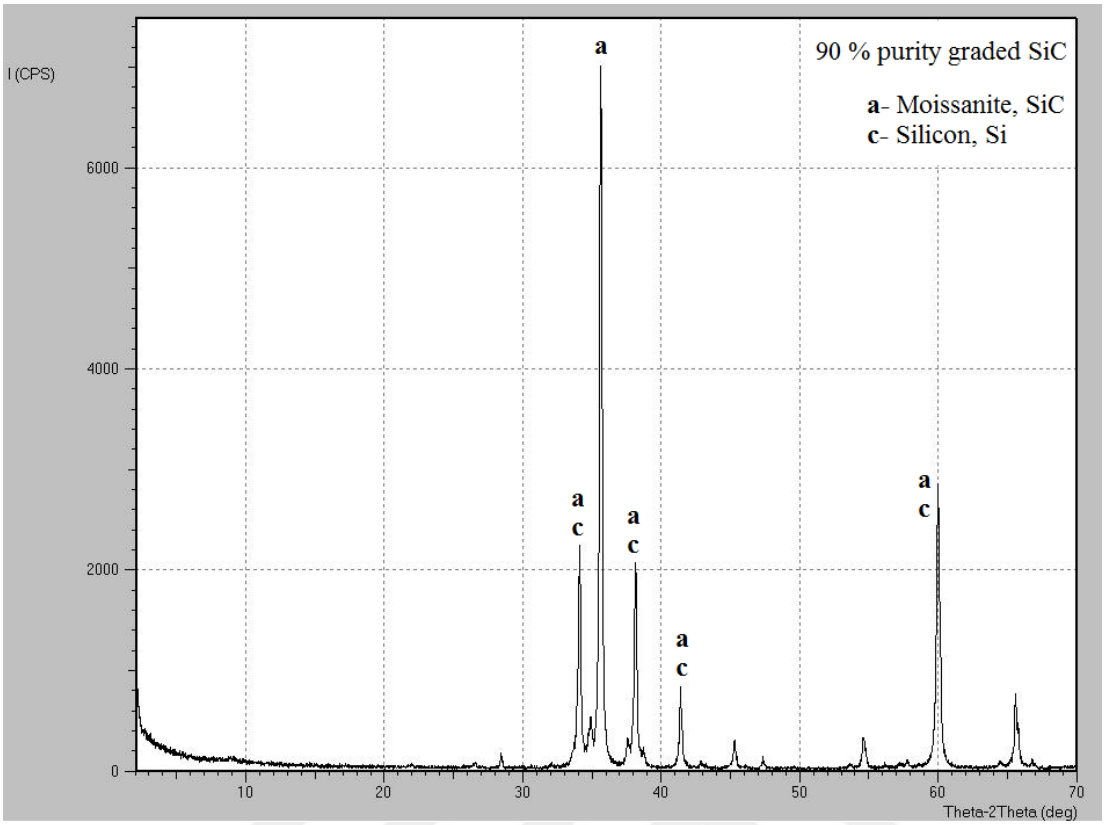


Figure 3.10. XRD pattern of 90 % purity graded SiC powders.

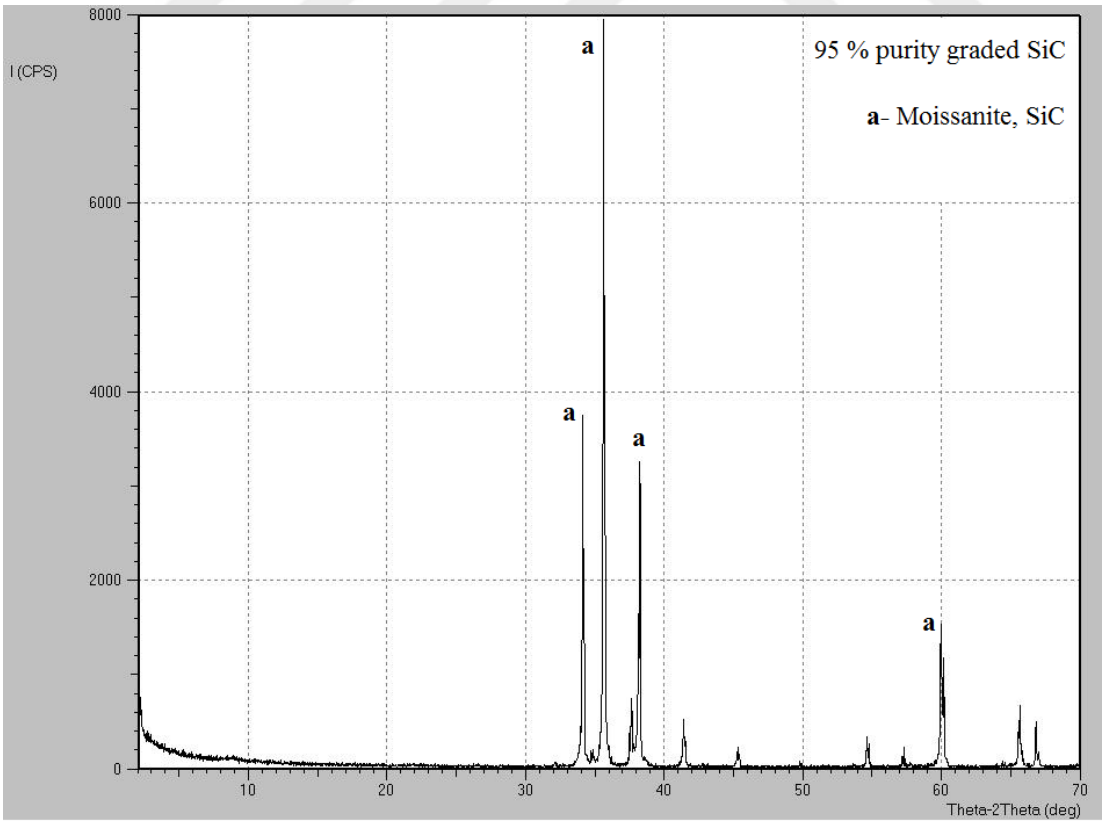


Figure 3.11. XRD pattern of 95 % purity graded SiC powders.

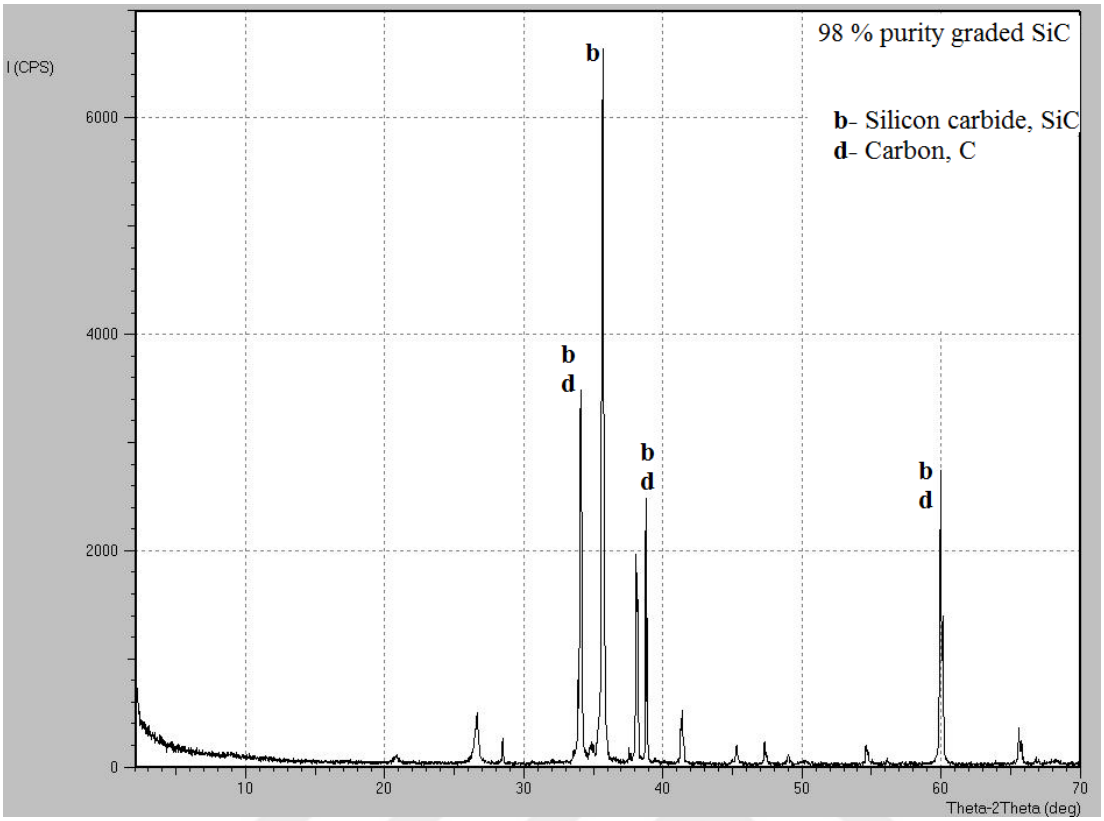


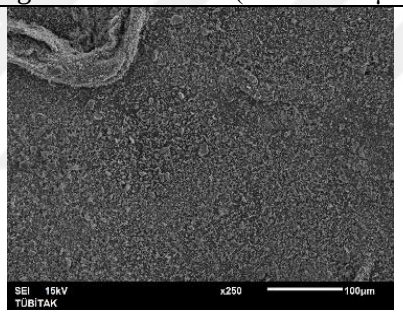
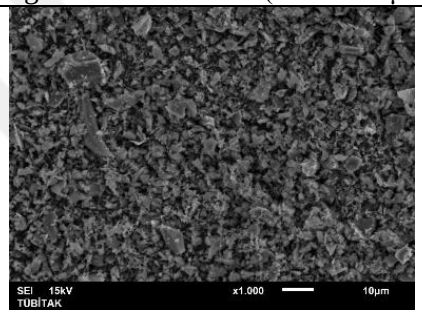
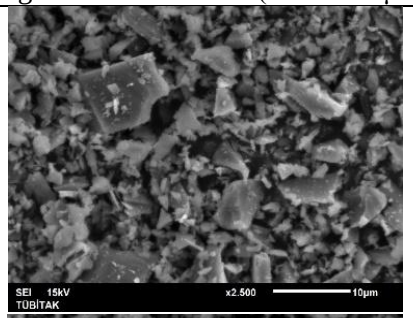
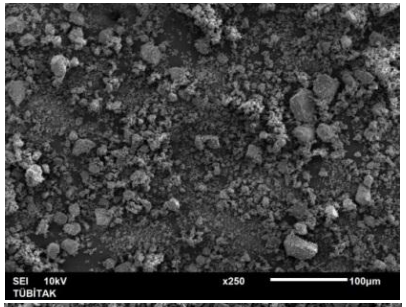
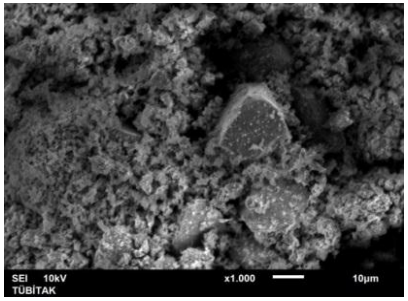
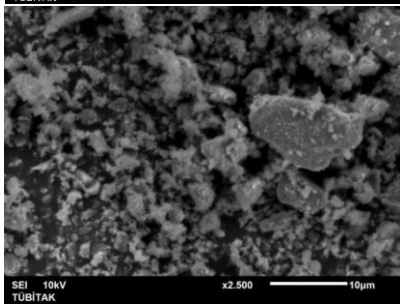
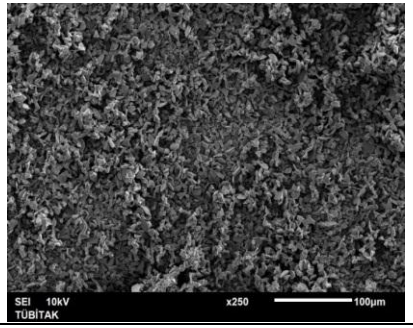
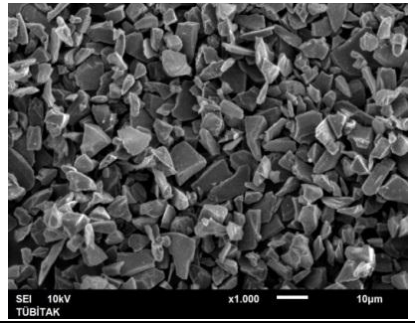
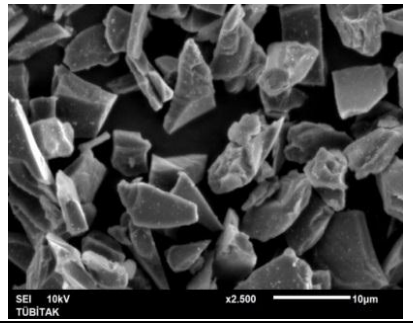
Figure 3.12. XRD pattern of 98 % purity graded SiC powders.

3.1.5. Scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) investigation of SiC raw materials

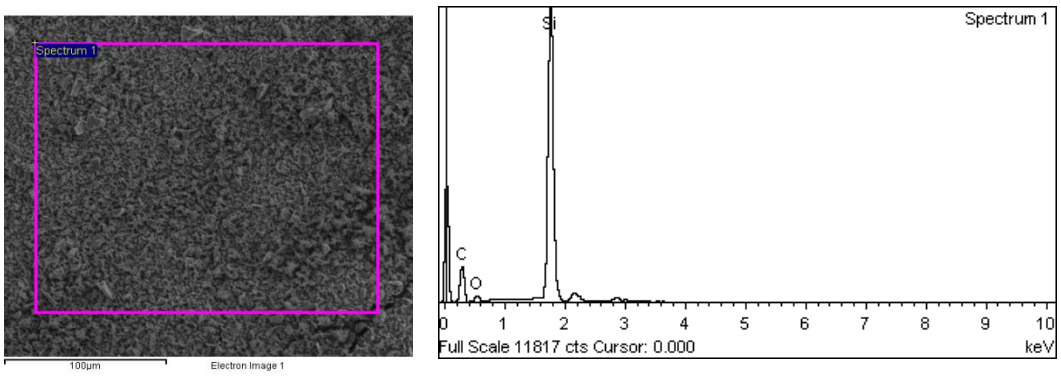
SEM investigation results of SiC powders were given in Table 3.5. The most homogenous, the sharpest and the smoothest surface area were observed at 98 % purity graded SiC when compared with other purity SiC grades. Also, it should be said that some inhomogeneous surface structures were significant at 95 % purity graded SiC. This was resulted from the grinding of 95 % purity graded SiC to decrease the grain size.

Moreover, energy dispersive spectrometer (EDS) analysis results were illustrated in Figure 3.13. The spectrum results supported the purity differences of SiC powders. The highest impurity was observed at 90 % purity graded SiC. Also, the highest atomic and weight percentage of Si-C ratio was measured at 98 % purity graded SiC powder.

Table 3.5. Microstructure images of SiC raw materials by using scanning electron microscope (SEM).

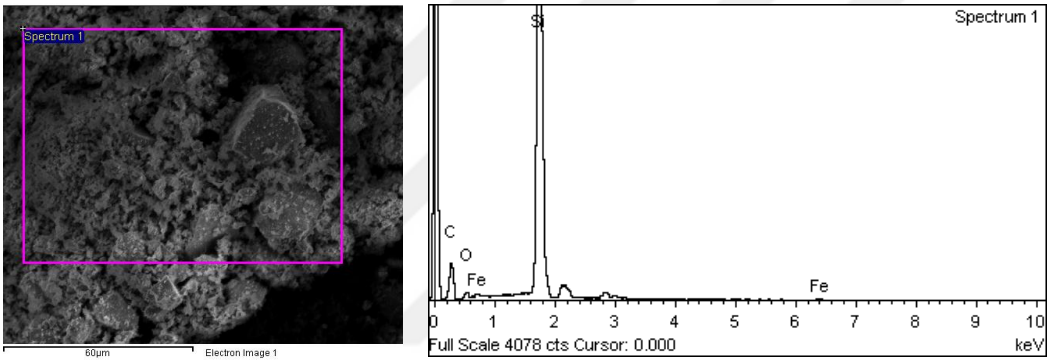
Sample Name	Magnification= x250 (Scale=100 µm)	Magnification= x1.000 (Scale=10 µm)	Magnification= x2.500 (Scale=10 µm)
SiC *(90 % purity graded)*			
SiC *(95 % purity graded)*			
SiC *(98 % purity graded)*			

(a) 90 % purity graded SiC powder



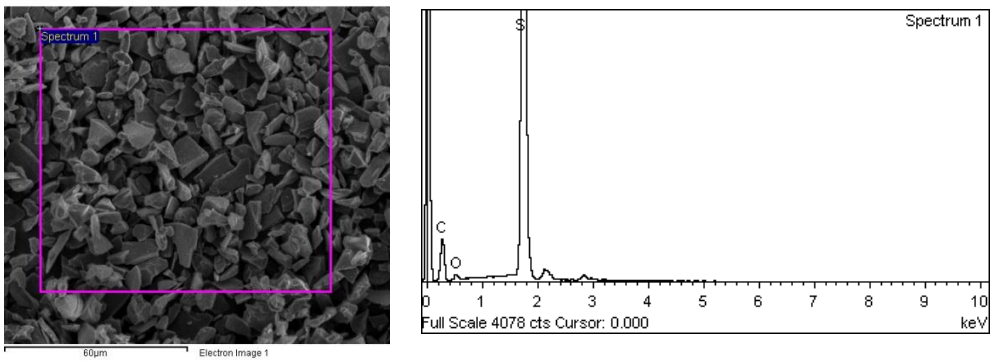
Element	C	O	Si	Total
Weight (%)	37.08	3.11	59.81	100.00
Atomic (%)	57.06	3.59	39.36	100.01

(b) 95 % purity graded SiC powder



Element	C	O	Si	Fe	Total
Weight (%)	29.10	2.01	66.51	2.39	100.00
Atomic (%)	48.85	2.53	47.75	0.86	99.99

(c) 98 % purity graded SiC powder



Element	C	O	Si	Total
Weight (%)	32.22	2.04	65.73	100.00
Atomic (%)	52.09	2.48	45.44	100.01

Figure 3.13. EDS analysis results of **(a)** 90 % purity graded SiC powder; **(b)** 95 % purity graded SiC powder and **(c)** 98 % purity graded SiC powder.

3.2. Foam Glass Samples

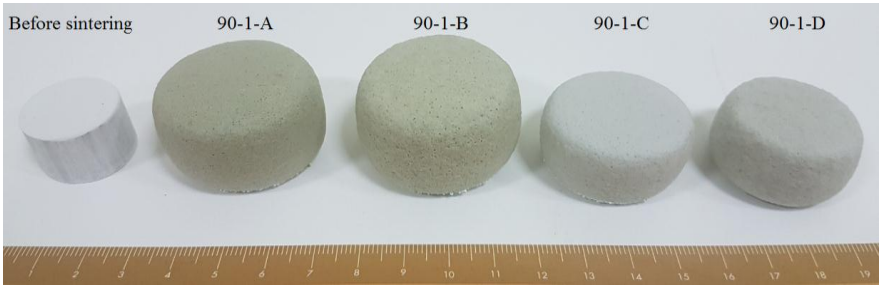
Upon completion sintering process, foam glasses with porous structure were obtained at all different temperature, composition and purity grades. Prior to determine the best formulas for desired properties, foam glass samples were given a code/sample number according to the their foaming agent purity grade, composition ratio and sintering temperature. These codes were useful for easy to compare. After sintering, details of foam glass sample codes and their fotographs were depicted in the following section.

3.2.1. 90 % Purity graded SiC foam glass samples

Explanation of the 90 % purity graded SiC foam glass codes were given in Table 3.6. Also, fotographs of these samples were shown in Figure 3.14 and 3.15 according to different composition ratios and different sintering temperatures.

Table 3.6. 90 % Purity graded SiC foam glass sample codes.

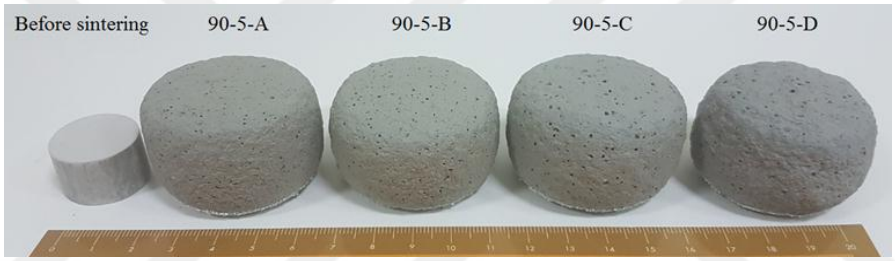
Sample codes	Composition		Sintering temperature (°C)
	SiC powder (*90 % purity*) (wt. %)	Glass powder (wt. %)	
90-1-A	1	99	800
90-3-A	3	97	800
90-5-A	5	95	800
90-7-A	7	93	800
90-9-A	9	91	800
90-1-B	1	99	825
90-3-B	3	97	825
90-5-B	5	95	825
90-7-B	7	93	825
90-9-B	9	91	825
90-1-C	1	99	850
90-3-C	3	97	850
90-5-C	5	95	850
90-7-C	7	93	850
90-9-C	9	91	850
90-1-D	1	99	875
90-3-D	3	97	875
90-5-D	5	95	875
90-7-D	7	93	875
90-9-D	9	91	875



(a) Samples before and after sintering at 800, 825, 850 and 875°C with 1 wt. % SiC (90 % purity)



(b) Samples before and after sintering at 800, 825, 850 and 875°C with 3 wt. % SiC (90 % purity)



(c) Samples before and after sintering at 800, 825, 850 and 875°C with 5 wt. % SiC (90 % purity)

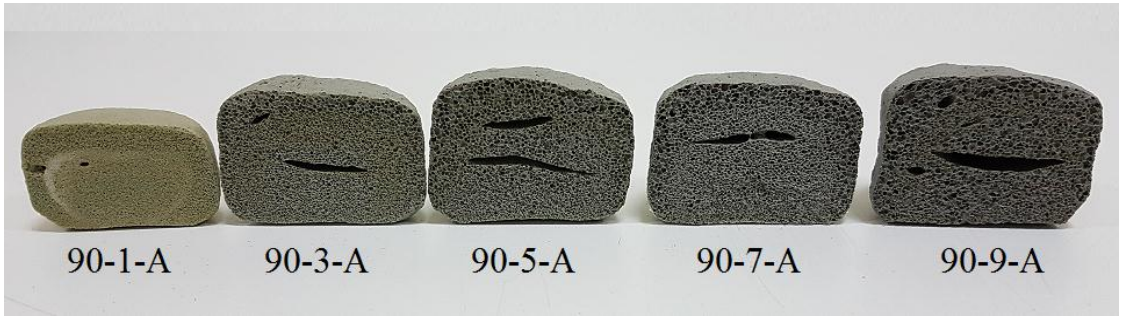


(d) Samples before and after sintering at 800, 825, 850 and 875°C with 7 wt. % SiC (90 % purity)

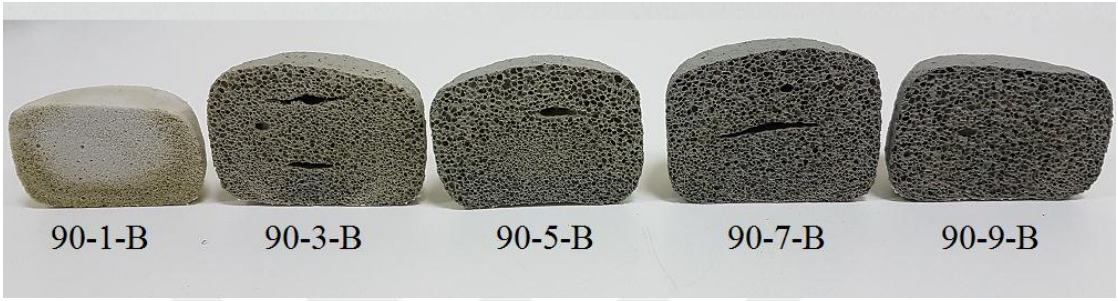


(e) Samples before and after sintering at 800, 825, 850 and 875°C with 9 wt. % SiC (90 % purity)

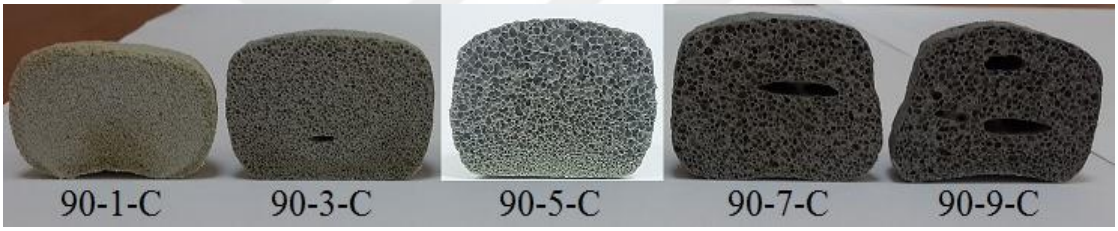
Figure 3.14. Illustration of foam glass samples with 90 % purity graded SiC addition before sintering and after sintering at 800, 825, 850 and 875°C with a ratio of **(a)** 1 wt.%; **(b)** 3 wt.%; **(c)** 5 wt.%; **(d)** 7 wt.%; **(e)** 9 wt.%, respectively.



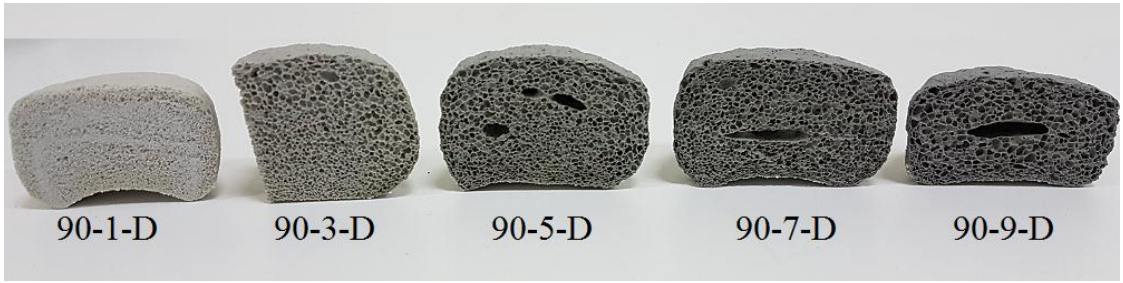
(a) Crosscut samples with 1, 3, 5, 7 and 9 wt.% SiC (90 % purity) addition fired at 800°C



(b) Crosscut samples with 1, 3, 5, 7 and 9 wt.% SiC (90 % purity) addition fired at 825°C



(c) Crosscut samples with 1, 3, 5, 7 and 9 wt.% SiC (90 % purity) addition fired at 850°C



(d) Crosscut samples with 1, 3, 5, 7 and 9 wt.% SiC (90 % purity) addition fired at 875°C

Figure 3.15. Illustration of crosscut foam glass samples with 90% purity graded SiC after sintering at **(a)** 800°C; **(b)** 825°C; **(c)** 850°C ; **(d)** 875°C, with a ratio of 1, 3, 5, 7 and 9 wt.%, respectively.

3.2.2. 95 % Purity graded SiC foam glass samples

95 % purity graded SiC containing foam glass codes were specified in Table 3.7. Moreover, figures of these samples were illustrated in Figure 3.16 and 3.17 according to different composition ratios and different sintering temperatures.

Table 3.7. 95 % Purity graded SiC foam glass sample codes.

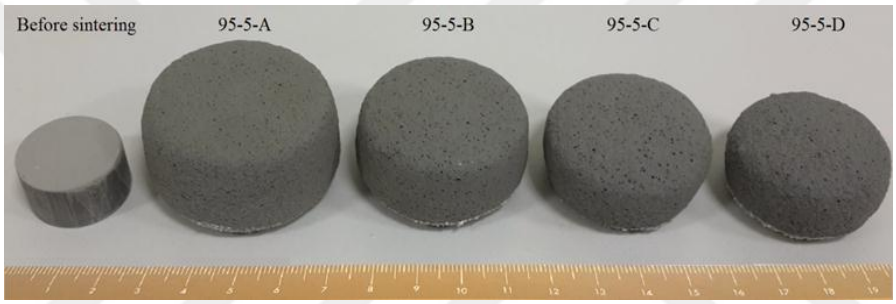
Sample codes	Composition		Sintering temperature (°C)
	SiC powder (*95 % purity*) (wt. %)	Glass powder (wt. %)	
95-1-A	1	99	800
95-3-A	3	97	800
95-5-A	5	95	800
95-7-A	7	93	800
95-9-A	9	91	800
95-1-B	1	99	825
95-3-B	3	97	825
95-5-B	5	95	825
95-7-B	7	93	825
95-9-B	9	91	825
95-1-C	1	99	850
95-3-C	3	97	850
95-5-C	5	95	850
95-7-C	7	93	850
95-9-C	9	91	850
95-1-D	1	99	875
95-3-D	3	97	875
95-5-D	5	95	875
95-7-D	7	93	875
95-9-D	9	91	875



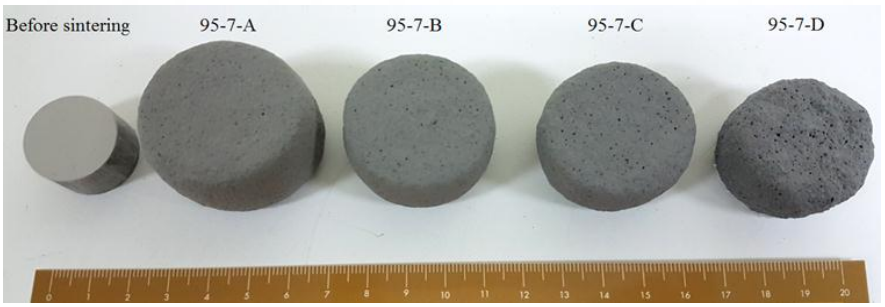
(a) Before and after sintering at 800, 825, 850 and 875°C with 1 wt. % SiC (95 % purity)



(b) Before and after sintering at 800, 825, 850 and 875°C with 3 wt. % SiC (95 % purity)



(c) Before and after sintering at 800, 825, 850 and 875°C with 5 wt. % SiC (95 % purity)



(d) Before and after sintering at 800, 825, 850 and 875°C with 7 wt. % SiC (95 % purity)

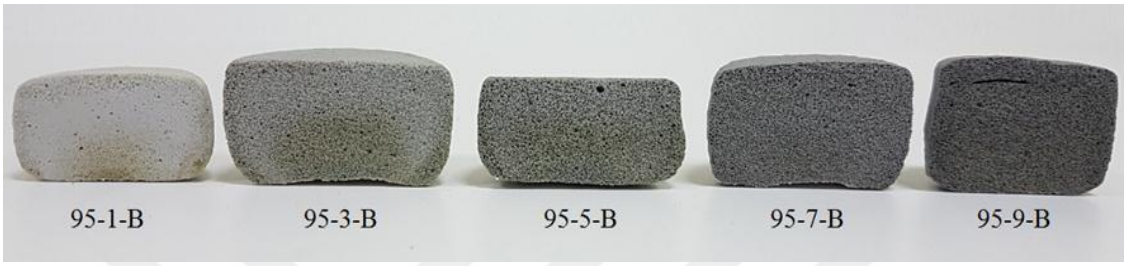


(e) After sintering at 800, 825, 850 and 875°C with 9 wt. % SiC (95 % purity).

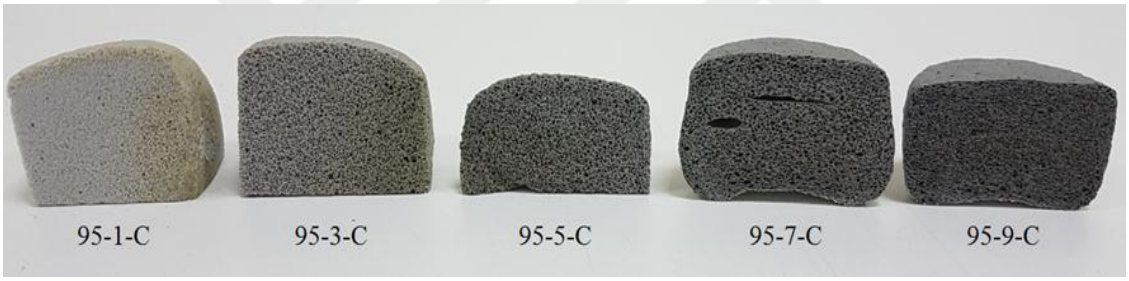
Figure 3.16. Illustration of foam glass samples with 95 % purity graded SiC addition before sintering and after sintering at 800, 825, 850, 875°C with **(a)** 1 wt.%; **(b)** 3 wt.%; **(c)** 5 wt.%; **(d)** 7 wt.%; **(e)** 9 wt.%, respectively.



(a) Crosscut samples with 1, 3, 5, 7 and 9 wt.% SiC (95 % purity) addition fired at 800°C



(b) Crosscut samples with 1, 3, 5, 7 and 9 wt.% SiC (95 % purity) addition fired at 825°C



(c) Crosscut samples with 1, 3, 5, 7 and 9 wt.% SiC (95 % purity) addition fired at 850°C



(d) Crosscut samples with 1, 3, 5, 7 and 9 wt.% SiC (95 % purity) addition fired at 875°C

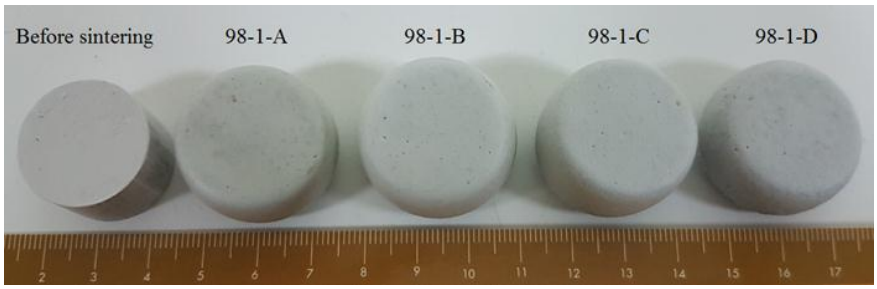
Figure 3.17. Illustration of crosscut foam glass samples with 95% purity graded SiC after sintering at **(a)** 800°C; **(b)** 825°C; **(c)** 850°C ; **(d)** 875°C, with a ratio of 1, 3, 5, 7 and 9 wt.%, respectively.

3.2.3. 98 % Purity graded SiC foam glass samples

98 % purity graded SiC containing foam glass codes were specified in Table 3.8 In addition, these samples views were shown in Figure 3.18 and 3.19 according to different composition ratios and different sintering temperatures.

Table 3.8. 98 % Purity graded SiC foam glass sample codes.

Sample codes	Composition		Sintering temperature (°C)
	SiC powder *(98 % purity)* (wt. %)	Glass powder (wt. %)	
98-1-A	1	99	800
98-3-A	3	97	800
98-5-A	5	95	800
98-7-A	7	93	800
98-9-A	9	91	800
98-1-B	1	99	825
98-3-B	3	97	825
98-5-B	5	95	825
98-7-B	7	93	825
98-9-B	9	91	825
98-1-C	1	99	850
98-3-C	3	97	850
98-5-C	5	95	850
98-7-C	7	93	850
98-9-C	9	91	850
98-1-D	1	99	875
98-3-D	3	97	875
98-5-D	5	95	875
98-7-D	7	93	875
98-9-D	9	91	875



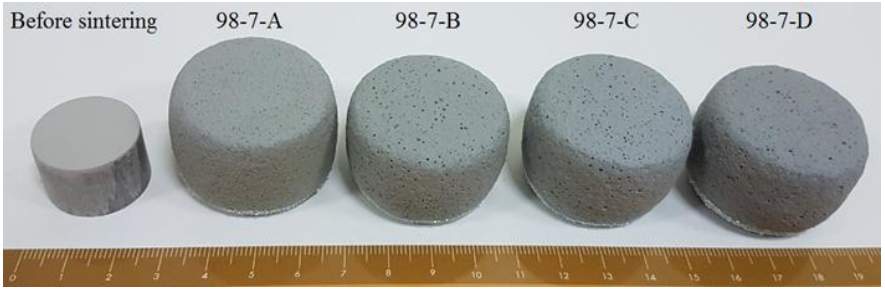
(a) Before and after sintering at 800, 825, 850 and 875°C with 1 wt. % SiC (98 % purity)



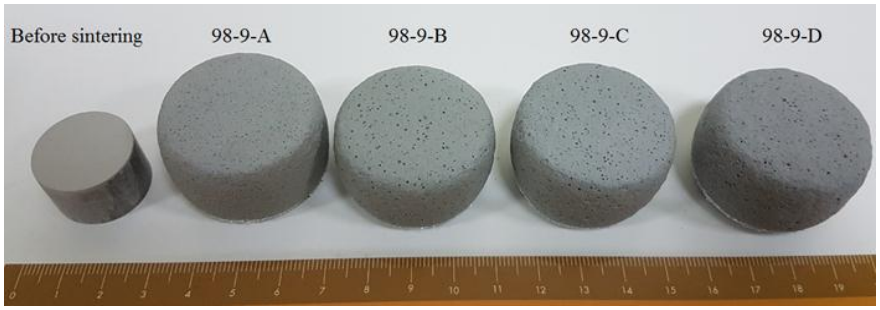
(b) Before and after sintering at 800, 825, 850 and 875°C with 3 wt. % SiC (98 % purity)



(c) Before and after sintering at 800, 825, 850 and 875°C with 5 wt. % SiC (98 % purity)



(d) Before and after sintering at 800, 825, 850 and 875°C with 7 wt. % SiC (98 % purity)



(e) Before and after sintering at 800, 825, 850 and 875°C with 9 wt. % SiC (98 % purity)

Figure 3.18. Illustration of foam glass samples with 98 % purity graded SiC addition before sintering and after sintering at 800, 825, 850 and 875°C with a ratio of **(a)** 1 wt.%; **(b)** 3 wt.%; **(c)** 5 wt.%; **(d)** 7 wt.%; **(e)** 9 wt.%, respectively.



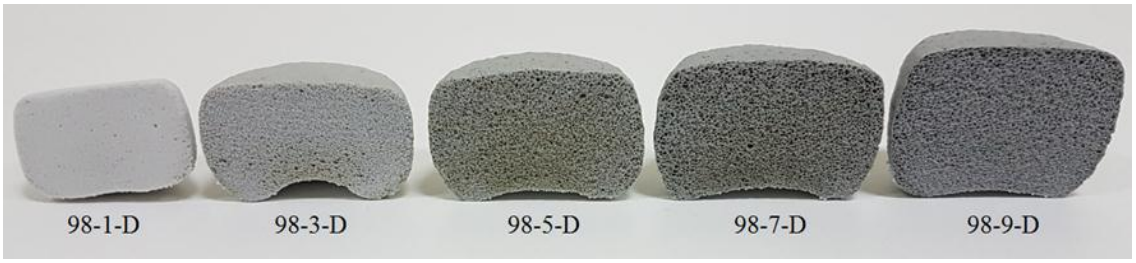
(a) Crosscut samples with 1, 3, 5, 7 and 9 wt.% SiC (90 % purity) addition fired at 800°C



(b) Crosscut samples with 1, 3, 5, 7 and 9 wt.% SiC (90 % purity) addition fired at 825°C



(c) Crosscut samples with 1, 3, 5, 7 and 9 wt.% SiC (90 % purity) addition fired at 850°C



(d) Crosscut samples with 1, 3, 5, 7 and 9 wt.% SiC (90 % purity) addition fired at 875°C

Figure 3.19. Illustration of crosscut foam glass samples with 98 % purity graded SiC after sintering at **(a)** 800°C; **(b)** 825°C; **(c)** 850°C ; **(d)** 875°C, with a ratio of 1, 3, 5, 7 and 9 wt.%, respectively.

3.3. Characterization Results and Discussion of Foam Glass Samples

3.3.1. Volume expansion ratios

As a result of foaming process, evolved gases cause a pressure within the viscous glass, then a large number of cells occur and lead to expansion of glass. Finally, macro porous foam glass material remains as volumetrically expanded body obviously. The more foaming reaction provides the more volume expansion. Based on the experimental studies, it was observed that volume expansion is one of the most important result to estimate the sufficiency of foam glass.

Actually, the extent of volume expansion gives an impression about the magnitude of porosity, density, mechanical strength and even thermal conductivity properties. Higher volume expansion leads large amount of pores due to entrapped gas bubbles within the body. This high porosity reduces the density of foam glass. In general, low density supplies lower thermal conductivity and enhances the thermal insulation. On the other hand, mechanical strength will be lower due to high porosity and low density caused by high amount of volume expansion.

Volume changes of samples were calculated by measuring of diameter (mm) and thickness (mm) of cyclindirical samples before and after sintering. It was determined that all foam glass samples were volumetrically expanded after sintering process. Volume expansion results were given in Table 3.9 for foam glass samples produced with various % purity graded SiC with different addition ratios and different sintering temperatures.

Table 3.9. Volume expansion ratios (%) of foam glass samples after firing.

SiC Purity Grade (%)	Temperature (°C)	SiC Addition Ratio (wt. %)				
		1	3	5	7	9
90	800	384.57	632.72	692.75	679.75	671.88
	825	413.74	711.37	759.57	726.71	746.81
	850	475.73	583.65	**802.48**	788.04	759.29
	875	426.48	634.79	681.40	603.94	566.93
95	800	233.55	454.46	522.35	554.53	519.90
	825	259.22	481.02	505.52	475.82	396.97
	850	270.38	359.98	375.40	390.38	311.04
	875	210.05	207.24	271.55	231.03	234.00
98	800	76.66	221.17	312.07	354.60	354.00
	825	118.96	246.26	304.82	342.61	384.15
	850	102.21	224.33	320.19	387.22	392.38
	875	112.18	233.00	311.29	361.93	415.84

The graphs of volume expansion results based on the foam glass samples produced with different temperatures, different foaming agent purity and addition ratio were given in Figure 3.20-3.22.

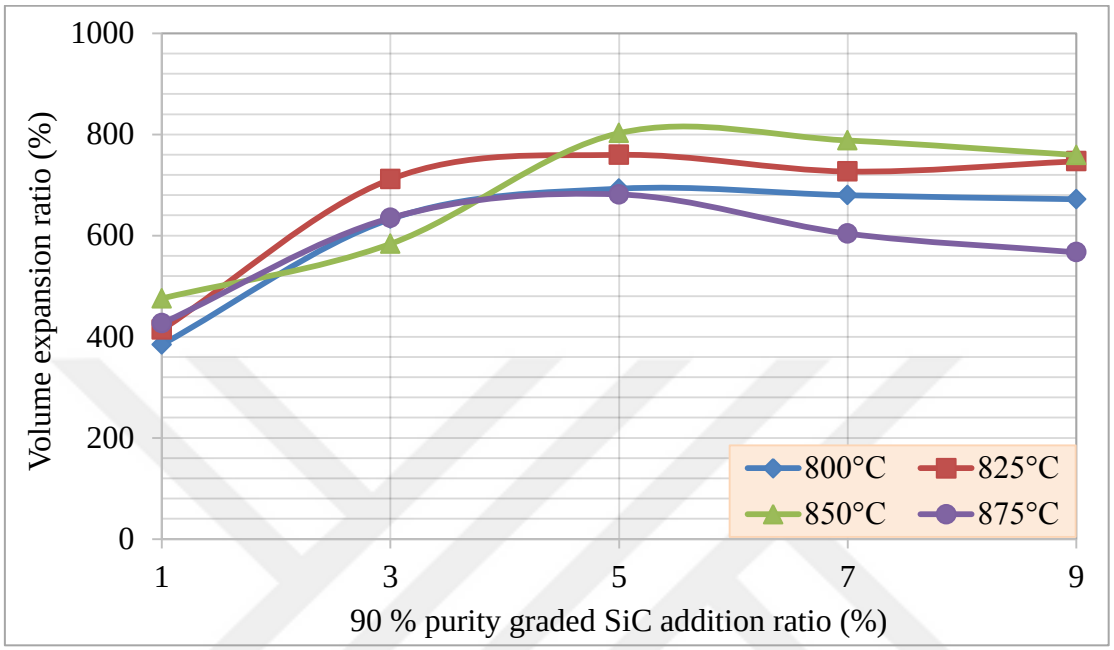


Figure 3.20. Volume expansion graph of foam glass samples with 90 % purity graded SiC with different addition ratios and different sintering temperatures.

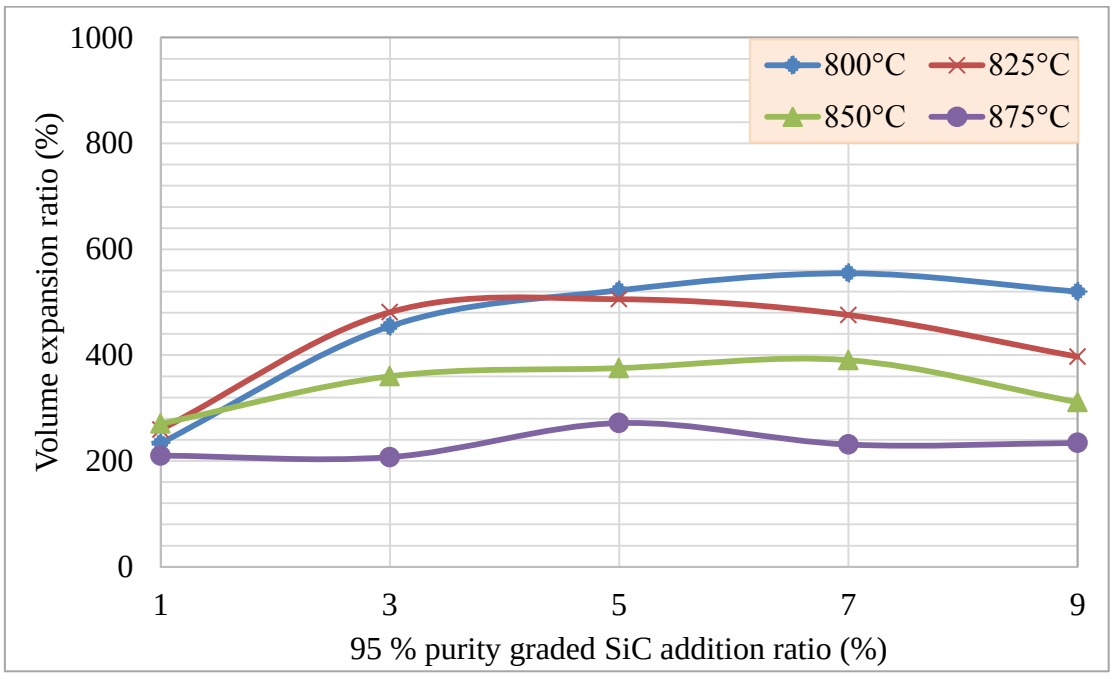


Figure 3.21. Volume expansion graph of foam glass samples with 95 % purity graded SiC with different addition ratios and different sintering temperatures.

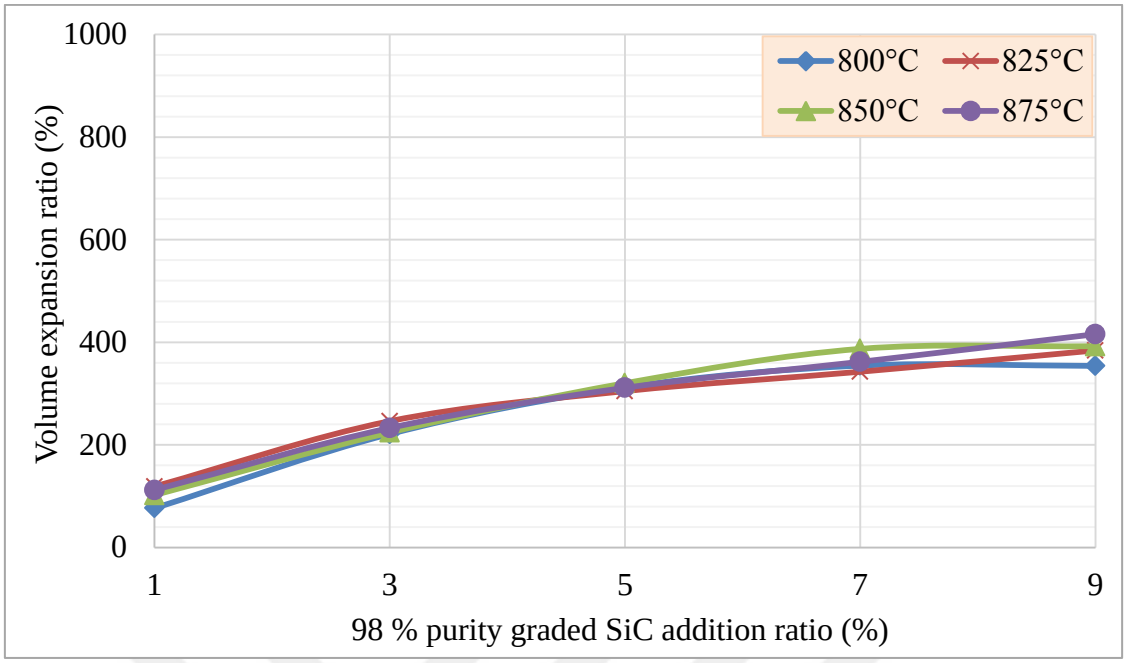


Figure 3.22. Volume expansion graph of foam glass samples with 98 % purity graded SiC with different addition ratios and different sintering temperatures.

According to the graphs, increasing of foaming agent ratio enhanced the volume expansion in all samples. 90 % purity grade SiC revealed the highest volume expansion with a ratio of 5 wt. % at 850°C. However, there was a reduction at volume expansion with addition of SiC (90% purity) above 5 wt. %. Similarly, 95 % purity grade SiC provide higher volume expansion with a ratio of 5 and 7 wt. %, however, slight declining of volume expansion was observed above the addition ratio of 7 wt. % SiC.

Gradually increasing of volume expansion was observed at 98 % purity graded SiC foam glass by the rising of 98 % purity graded SiC addition at all firing temperatures. Even though this gradually increasing volumetric expansion, the lowest volume expansion values was also observed at 98 % purity graded SiC foam glass among all samples with various SiC purity grades This can be attributed to the higher melting point of SiC as the purity of SiC rises and higher temperature requirements for foaming reaction with higher purity grade of SiC. Thus, better foaming behaviors and products were achieved with 90 % purity graded SiC foam glasses which were easily foamed at the same sintering temperatures when compare with 95 and 98 % purity graded SiC.

Temperature differences did not give significant results in terms of volume expansion because intervals of firing temperature between sintering temperature degrees seemingly very narrow.

3.3.2. Mineralogical analysis (XRD)

Cristobalite (SiO_2), moissanite (SiC), diopside ($Ca(Mg,Al)(Si,Al)_2O_6$) and akermanite ($Ca_2MgSi_2O_7$) phases were observed by XRD analysis for all foam glass samples. It was specified in literature that the release of silicon oxides from the foaming agents may result in partial crystallization of glass with precipitation of cristobalite [5]. Mineralogical analysis of this study proved that silicon oxides are released due to foaming reactions by the addition of SiC foaming agents. The intensity of the cristobalite peaks increased with increasing SiC foaming agent addition. Figure 3.23 shows that the highest cristobalite peak intensity occurred at the foam glass produced with 9 wt. % SiC (90% purity); on the other hand, the lowest cristobalite peak intensity was observed with 1 wt. % SiC (90% purity). In similar, increase in temperature and foaming agent purity grade both have an effect on intensity of cristobalite peaks (Figure 3.24). Since the foaming behavior was driven and controlled by temperature increase, cristobalite peak intensities enhanced by increasing temperature. Moreover, foaming activities were conducted easily with using 90 % purity grade SiC, similarly, higher cristobalite peak intensity was seen at 90% purity graded SiC foam glass. When SiC purity increased, foaming process became slower at the same firing temperatures, thus less reaction occurred and less cristobalite phase precipitated (Figure 3.25).

Also, unreacted moissanite (SiC) phase was observed at all foam glass samples because of residual foaming agent. This result is in a good agreement with the reported and confirmed results in the literature [68]. Since SiC has very high melting point (≈2730°C), it would be expected that there will be residual and unreacted SiC phases at relatively lower sintering temperature (800-875°C).

In addition, new crystalline phases like akermanite and diopside formed that it may be occured from reactions between impurities of SiC and waste glass and crystallization of the glass which take place during sintering. It was also observed that rising SiC content lead an increasing of akermanite and diopside peaks intensity. Similarly, these crystalline phase formed was more dominant at the samples which foaming was obtained at higher degree. Mineralogical phases and chemical formulas of foam glass samples after sintering were given in Table 3.10 with JCPDS (Joint Committee on Powder Diffraction Standards) numbers. To specify the effect of foaming agent purity

grades, composition ratios and sintering temperatures on phase development, patterns were overlaid according to the investigated parameters. XRD patterns of selected foam glass samples are shown in Figure 3.23-3.25.

Table 3.10. Mineralogical phases detected after firing of foam glass samples.

Mineralogical Phase	Chemical Formula	JCPDS #
Cristobalite	SiO_2	39-1425
Moissanite	SiC	29-1131
Diopside	$Ca(Mg,Al)(Si,Al)_2O_6$	41-1370
Akermanite	$Ca_2MgSi_2O_7$	35-592
Amorphous		

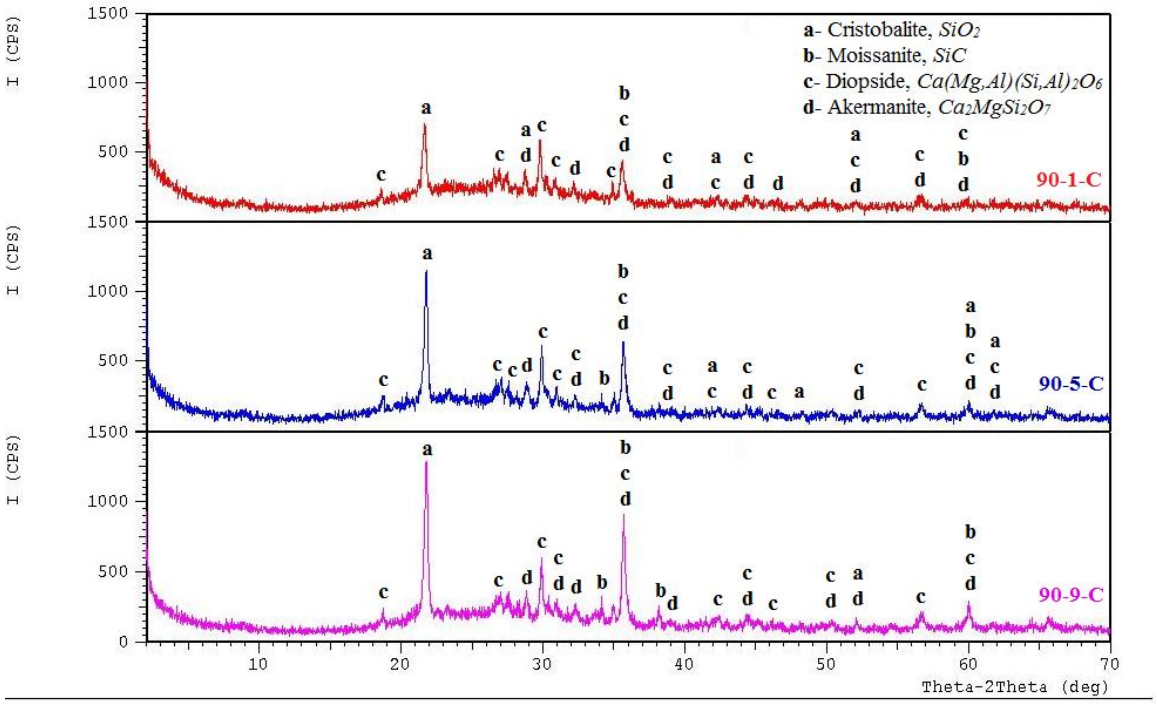


Figure 3.23. Mineralogical phase patterns of foam glass with 1, 5 and 9 wt. % SiC (90 % purity) and sintered at 850°C (90-1-C, 90-5-C and 90-9-C, respectively).

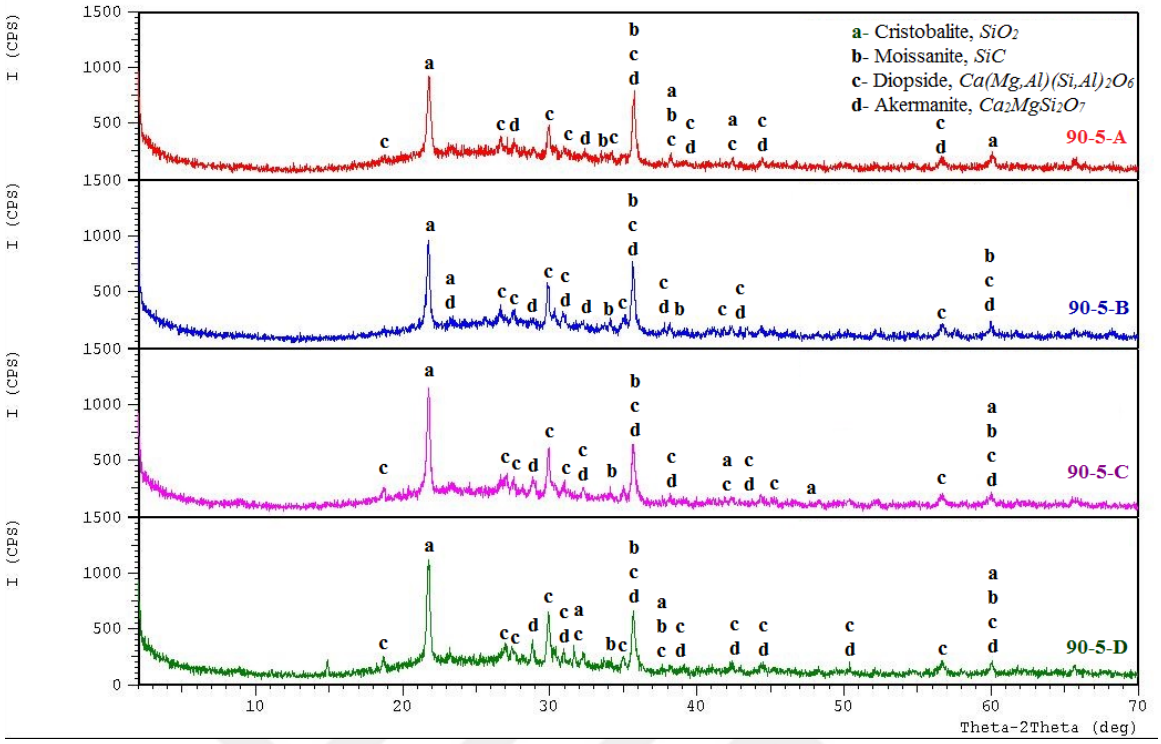


Figure 3.24. Mineralogical phase patterns of foam glass with 5 wt. % SiC (90 % purity) and sintered at 800, 825, 850 and 875°C (90-5-A, 90-5-B, 90-5-C and 90-5-D, respectively).

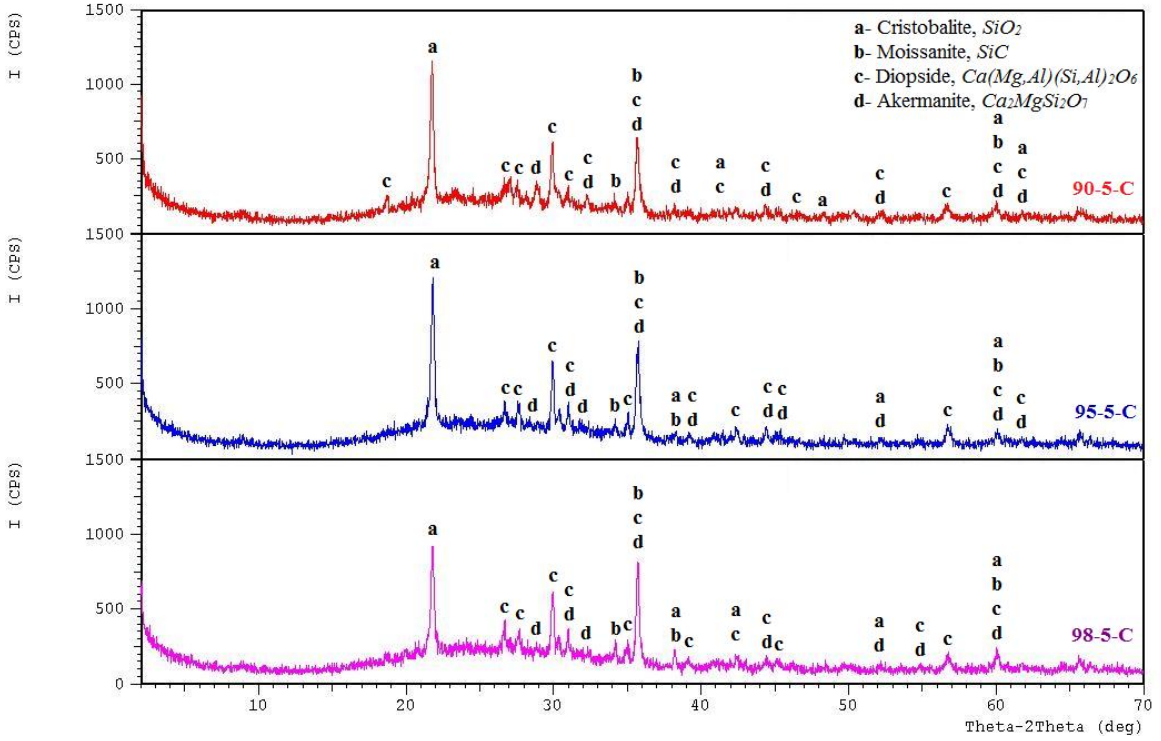


Figure 3.25. Mineralogical phase patterns of foam glass with 5 wt. % SiC (90, 95 and 98 % purity) and sintered at 850°C (90-5-C, 95-5-C and 98-5-C, respectively).

3.3.3. Compressive strength

Cellular morphology of foam glass provides substantial compressive strength. However, it must be said that foam glass products cannot be used as bearing materials in buildings. They are used for between the construction walls or around the zone to be insulated as an insulation material. Thus, high level compressive strength is not mandatory but foam glass should be rigid and ensure at least ≥0.4 – 3 MPa according to the standard TS EN 13167:2013-04 [24]. Compressive strength average results of foam glass samples were given in Table 3.11.

Table 3.11. Average compressive strength (MPa) values of foam glass samples.

SiC Purity Grade (%)	Temperature (°C)	SiC Addition Ratio (wt. %)				
		1	**3**	**5**	**7**	**9**
90	800	4.58	2.14	2.18	2.14	1.95
	825	5.25	2.01	1.70	1.75	1.56
	850	4.23	2.33	1.50	1.49	1.23
	875	4.03	1.41	1.72	1.61	1.34
95	800	15.39	5.88	3.83	3.51	3.81
	825	13.21	4.70	4.42	4.57	3.96
	850	8.24	3.17	2.61	2.94	4.17
	875	9.33	6.61	4.15	2.55	3.11
98	800	48.98	14.72	10.10	7.36	7.04
	825	23.13	14.30	10.93	8.03	6.19
	850	27.76	12.38	8.38	5.55	6.07
	875	23.07	13.28	6.99	4.42	4.76

In general, the reason of low mechanical strength of foam glass can be attributed to the large volumetric changes due to polymorphic transformation at cooling stage which can raise micro cracking within the cells [5].

From the Table 3.11, it was evident that foaming agent addition ratio, sintering temperature and even foaming agent purity grade affected compressive strength of foam glass. In addition, these parameters have considerable effects on mechanical strength as shown in Figure 3.26-3.28.

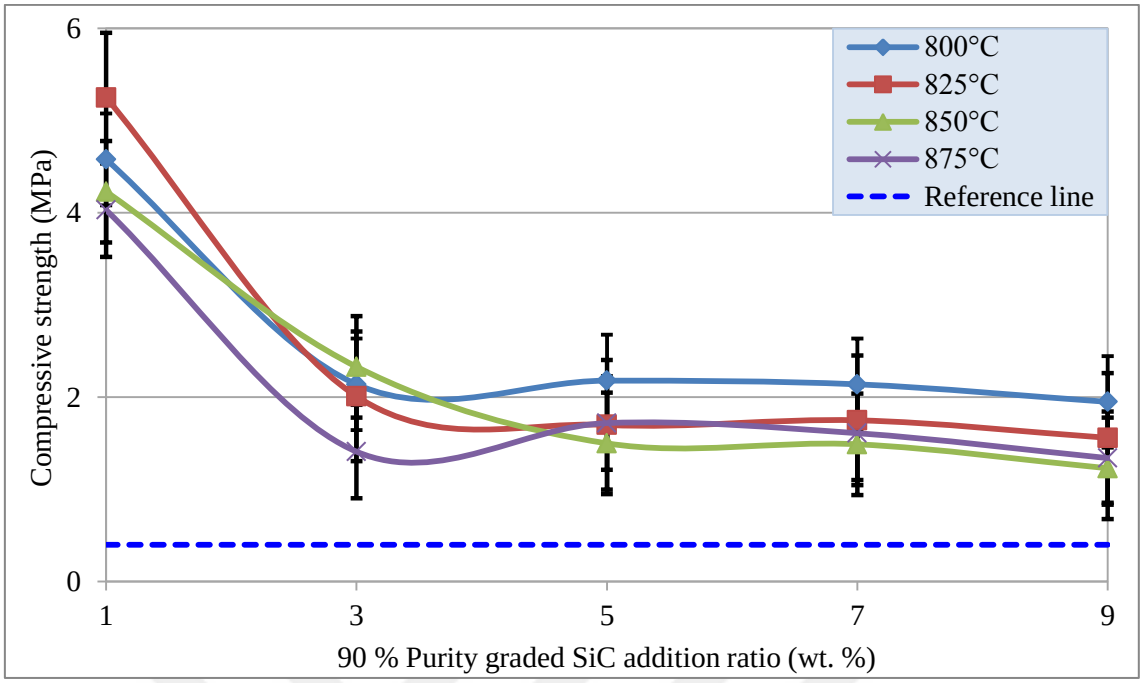


Figure 3.26. Compressive strength graph of foam glass samples with 90 % purity graded SiC with various addition ratios and sintering temperatures.

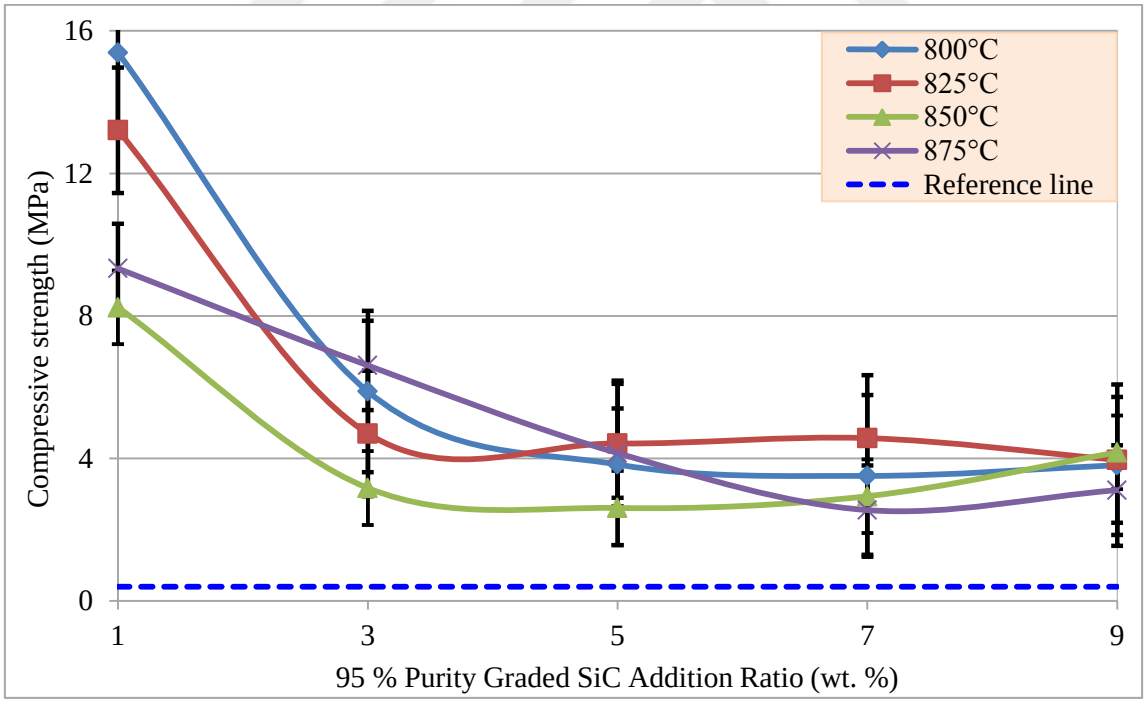


Figure 3.27. Compressive strength graph of foam glass samples with 95 % purity graded SiC with various addition ratios and sintering temperatures.

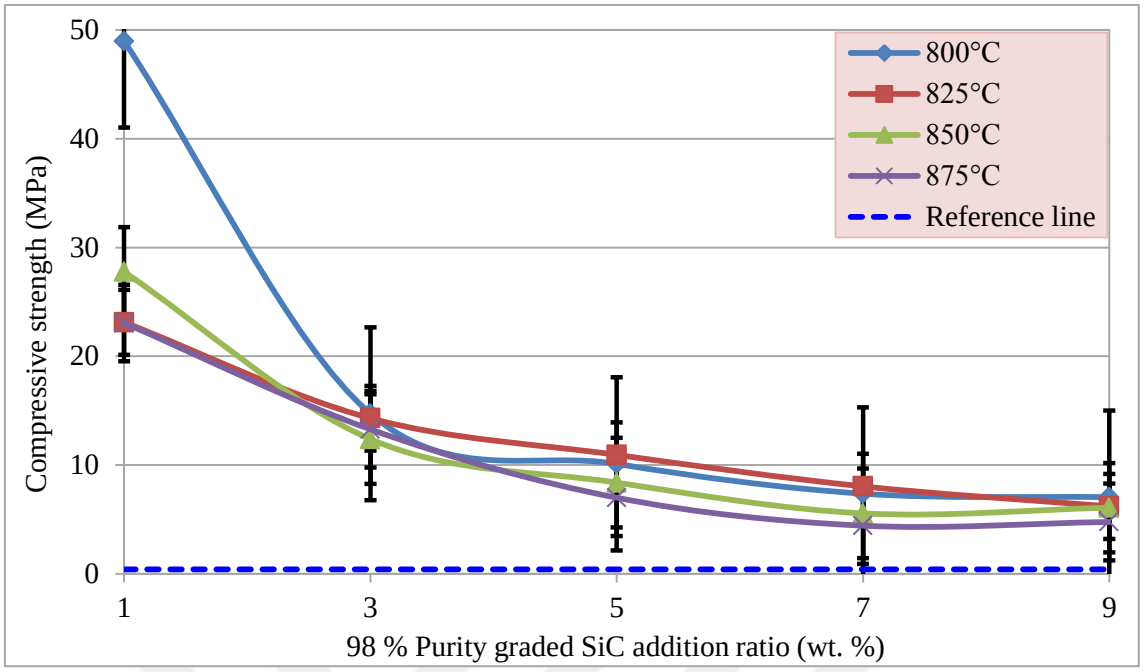


Figure 3.28. Compressive strength graph of foam glass samples with 98 % purity graded SiC with various addition ratios and sintering temperatures.

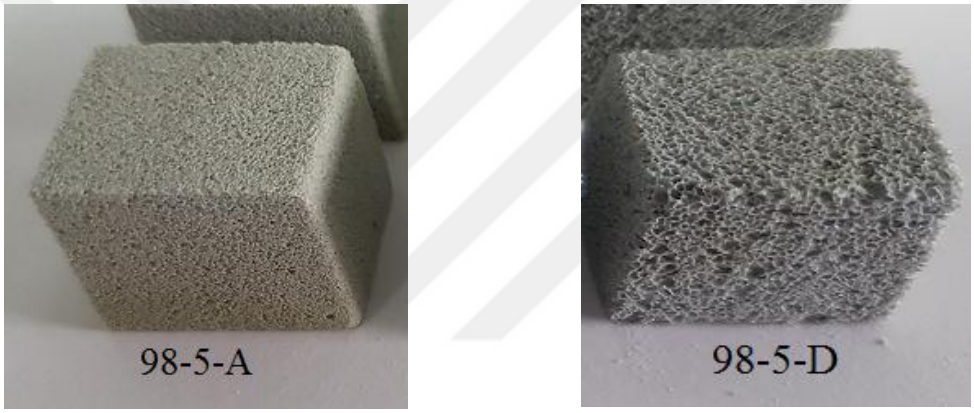
With increasing of foaming agent addition ratio, foaming process accelerated and generating cells enlarged resulted in strength reduction. These phenomena were seen at all foam glass samples. As it can be seen in the Figure 3.29, cell sizes increased at higher amount of foaming agent addition. Due to the fact that using of foaming agent with a ratio of 1 wt. % (90-1-D) provided smaller cells and higher compressive strength (4.03 MPa), while 9 wt. % foaming agent caused relatively higher cell size and lower compressive strength (1.34 MPa). Thus, it can be said that high amount of foaming agent negatively affect the foam glass with regard to compressive strength.



(a) **(b)**

Figure 3.29. Comparison of foaming agent addition effects on cell size for fired foam glass samples with a ratio of **(a)** 1 wt. % and **(b)** 9 wt. %.

Another factor of strength reduction may result from sintering at higher temperature. During temperature increase, initially smaller cells continue to grow or combine with each other because of the foam glass structure has sufficient viscosity and softness at these temperatures. When foaming agent purity grade and composition ratio parameters were constant but only sintering temperature was different, it was observed that all foam glass samples sintered at higher temperature had higher cell size, on the other hand, smaller cells were obtained from the samples sintered at lower temperatures as shown in Figure 3.30. When the foam glass with using 98 % purity graded SiC with a ratio of 5 wt. % sintered at 800°C, initial cell size was smaller and compressive strength was achieved as 10.0 MPa; on the other side, this sample were sintered at 875°C, cells enlarged and strength reduced to 6.99 MPa.

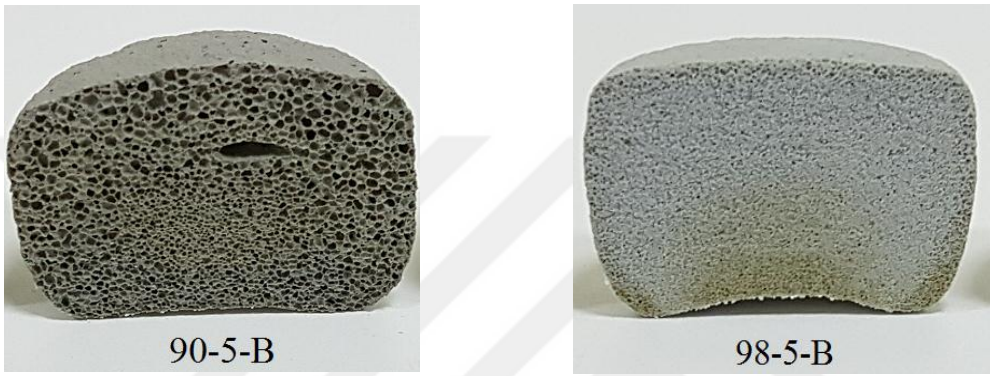


(a) **(b)**

Figure 3.30. Comparison of sintering temperature effects on cell size for fired foam glass samples having same composition which sintered at **(a)** 800°C and **(b)** 875°C.

SiC foaming agent purity grade, on the other hand, affected to the foaming reaction, cell size distribution and correspondingly compressive strength. It was observed that compressive strength of foam glass significantly increased with increasing SiC purity grade. One of the reasons of compressive strength increase is that higher purity graded SiC is harder and may strengthen the final foam glass products. Another reason of compressive strength increasing with high purity graded SiC may similarly be caused by smaller cell sizes. With increasing SiC purity, smaller cells occurred within the foam glass structure, while lower purity graded SiC caused larger cell distribution when samples were produced with using same composition and sintered at the same temperature. Cell size differences proceeded from lower purity grade SiC has lower melting point and can lead foaming reaction with glass easier rather than higher purity

grade SiC which has higher melting point and hardly react at the same temperature. In other words, 90 % purity graded SiC could foamed the glass faster, and evolved gaseous species and surrounded cells enlarged during reaction. However, 98 % purity graded SiC could react with glass later, and foaming process completed without time for developing cell size. Figure 3.31 shows the effect of SiC purity grade on compressive strength, 90 % purity graded SiC provides 1.70 MPa, while 98 % purity graded SiC could supply 10.93 MPa with using the same composition and sintered at the same temperature.



(a) **(b)**

Figure 3.31. Comparison of foaming agent purity effects on cell size for fired foam glass samples with **(a)** 90 % purity graded SiC and **(b)** 98 % purity graded SiC.

When compressive strength results of the all foam glass samples were compared, the foaming agent purity grade, composition ratio and sintering temperature effects on mechanical strength were proved. Maximum compressive strength was measured as 48.98 MPa from the sample with using 98 % purity graded SiC with a ratio of 1 wt. % and sintered at 800°C, in other words, the highest purity graded SiC, the lowest addition ratio of SiC foaming agent and the lowest sintering temperature revealed the highest compressive strength among all samples studied. Nevertheless, it also means that this highest strength resulted from the low degree foamed samples which may not supply the desired foam glass properties. Moreover, the weakest foam glass was achieved from the sample produced with using 90 % purity graded SiC with a ratio of 9 wt. % and sintered at 850 and 875°C. In other words, the lowest purity graded of SiC, the highest addition ratio of SiC foaming agent and the highest sintering temperature caused strength reduction due to the reasons of explained above.

It can be concluded that all foam glass samples produced in this study satisfied the

desired compressive strength. According to the required properties and the usage area, the starting formulation and raw materials should be chosen.

3.3.4. Apparent density

Light weight is one of the most important properties of foam glass products. As mentioned before, the desired apparent density is between 0.09 – 0.18 g/cm^3 according to the standard TS EN 13167:2013-04 [24]. Light weight comes from these extremely low densities, which is caused by the highly porous structure of foam glass.

As a result of foaming behavior during sintering, samples containing glass and foaming agent mixture transformed into cellular form and gaseous products revealing porous structure rather than sintered dense body. Before the sintering process, apparent density of the pressed foam glass samples (green products) were between 1.50 – 1.58 g/cm^3; after sintering process however, the density of foam glass samples dramatically decreased as given in Table 3.12.

Table 3.12. Average apparent density (g/cm^3) results of foam glass samples.

SiC Purity Grade (%)	Temperature (°C)	SiC Addition Ratio (wt. %)				
		1	3	5	7	9
90	800	0.290	0.195	0.182	0.184	0.184
	825	0.277	**0.177**	**0.169**	**0.178**	**0.176**
	850	0.251	0.208	**0.164**	**0.166**	**0.174**
	875	0.275	0.197	0.188	0.210	0.219
95	800	0.415	0.262	0.244	0.226	0.243
	825	0.392	0.249	0.248	0.256	0.297
	850	0.371	0.309	0.304	0.293	0.388
	875	0.466	0.445	0.404	0.452	0.454
98	800	0.710	0.443	0.360	0.318	0.310
	825	0.645	0.409	0.356	0.327	0.297
	850	0.669	0.438	0.358	0.296	0.291
	875	0.677	0.442	0.360	0.325	0.307

The primary reason of the density decrease after sintering is caused by volume expansion and the resultant porosity formation in the samples as shown in Figures 3.14-3.18. According to the foaming agent (SiC) purity grade, addition ratio and sintering

temperature, each of the shaped foam glass samples involved in foaming reaction in a different level. Some of them revealed high level of foam formation and volumetrically expanded up to 800 %, but others hardly foamed and minor expansion was obtained as mentioned in the previous sections. These differences in expansion level led to variation in cellular structures and in density.

The apparent density results of foam glass samples can be classified according to foaming agent purity grade. In the foam glasses with 90 % purity graded SiC, apparent density values reduced by foaming agent with a ratio of 5 wt. %, after then, an increase was observed at density values above 5 wt. % SiC addition ratio. Similarly, temperature increase from 800°C to 850°C provided density reduction, but after 850°C to 875°C caused an increase on the density values. These results corresponded to volume expansions of foam glasses. Especially samples exhibit high expansion rates provided lower density; when volumetric expansion decreased with above 5 wt. % SiC addition and at 850°C temperature, density values began to rise. Finally, it can be said that 5 wt. % SiC addition ratio and 850°C temperature are optimum parameters for 90 % purity graded SiC foam glass samples. The apparent density results of 90 % purity graded SiC foam glasses were graphically shown in Figure 3.32.

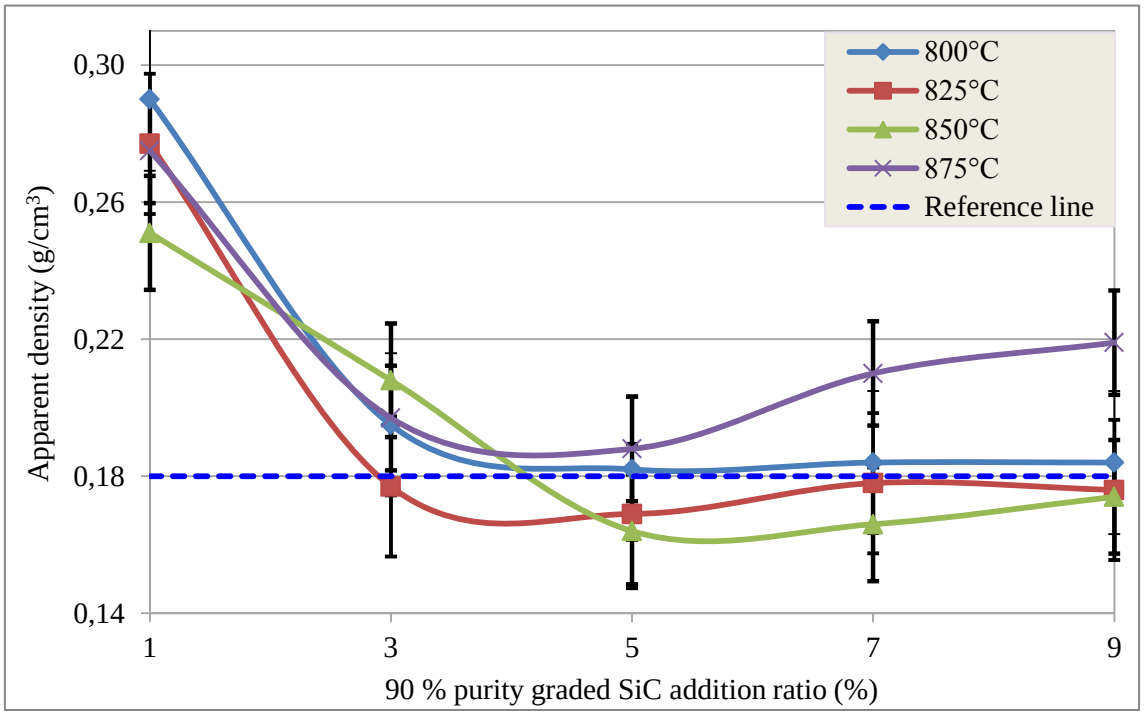


Figure 3.32. Apparent density variation of 90% grade SiC addition ratio of foam glass samples.

In 95 % purity graded SiC foam glasses, the lowest apparent density (g/cm^3) values were obtained with addition of 5 or 7 wt. % foaming agent. As it can be seen from the Figure 3.33, densities of the samples were higher below the 5 wt. % SiC and above 7 wt. % SiC addition. This is because SiC (95 % purity) provided the highest volumetric expansion and porosity with 5 and 7 wt. % addition, thus highly porous structures caused a visible reduction on the apparent density. On the other hand, increase in sintering temperature negatively affected the density of 95 % purity graded SiC foam glasses. As shown in the Figure 3.16 and Figure 3.17, foam glasses began to collapse and melt through temperature increasing from 800 to 875°C for 7 and 9 wt. % SiC containing samples. Above the 825°C temperature, expanded pores collapsed and cohered with each other, thus density began to rise with these inhomogeneous cellular bodies. Especially at the samples sintered at 875°C, collapsing and distortion of pores and melting of external zones of the samples can be easily seen. Because of the heat transforms from the outer side to the inside zone, shape distortion and melting began at the external surface firstly. Initially smaller cells of external surface as in the lower temperatures began to enlarge and struts of the cells (cell walls) thickened with increasing temperature to the 875°C. Thus, the highest density values of 95 % purity graded SiC foam glass samples were observed at sintered at 875°C.

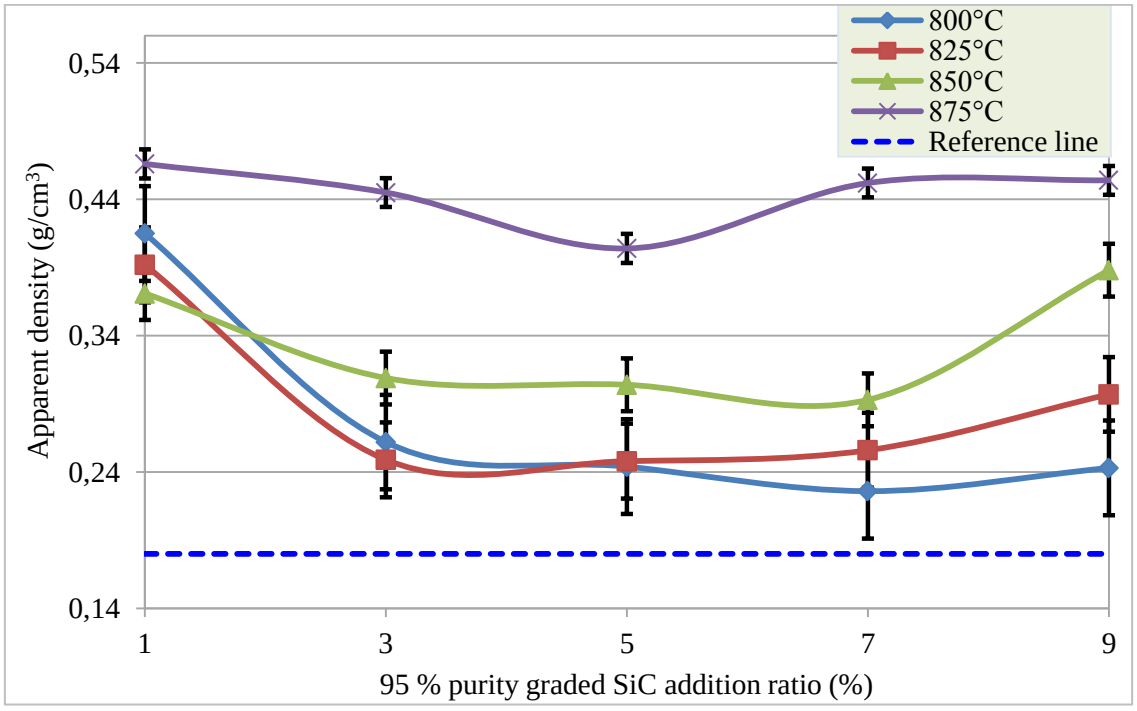


Figure 3.33. Apparent density variation of 95% grade SiC addition ratio of foam glass samples.

Foaming agent addition ratio effect can be definitely observed at 98 % purity graded SiC additions. Apparent density values of the samples continuously declined with addition of 98 % purity graded SiC from 1 wt. % to 9 wt. %. As depicted in Figure 3.18 and Figure 3.19, the samples produced with 1 wt. % SiC (98 % purity) slightly foamed, and samples revealed almost sintered glass texture. Thus, the highest density values were measured at these almost nonporous foam glass with 1 wt. % SiC (98 % purity). By increasing the foaming agent addition, foaming reactions could easily make a progress and satisfactory homogenous cell structure formed a significant decline on the density. Despite the continuously decreasing of density values, the lowest density of the 98 % purity graded SiC foam glasses was still higher according to the 90 and 95 % purity graded SiC foam glasses. This may cause from high purity SiC which has also higher density than others.

Nevertheless, temperature increase could not provide a significant effect on the density of 98 % purity graded SiC foam glasses. It may result from the higher melting point of 98 % purity graded SiC which is much higher than 875°C, thus foaming behaviors at the temperatures from 800°C to 875°C were close to each other. The effects of foaming agent (98 % purity grade) addition ratio and sintering temperature on the density were given in Figure 3.34.

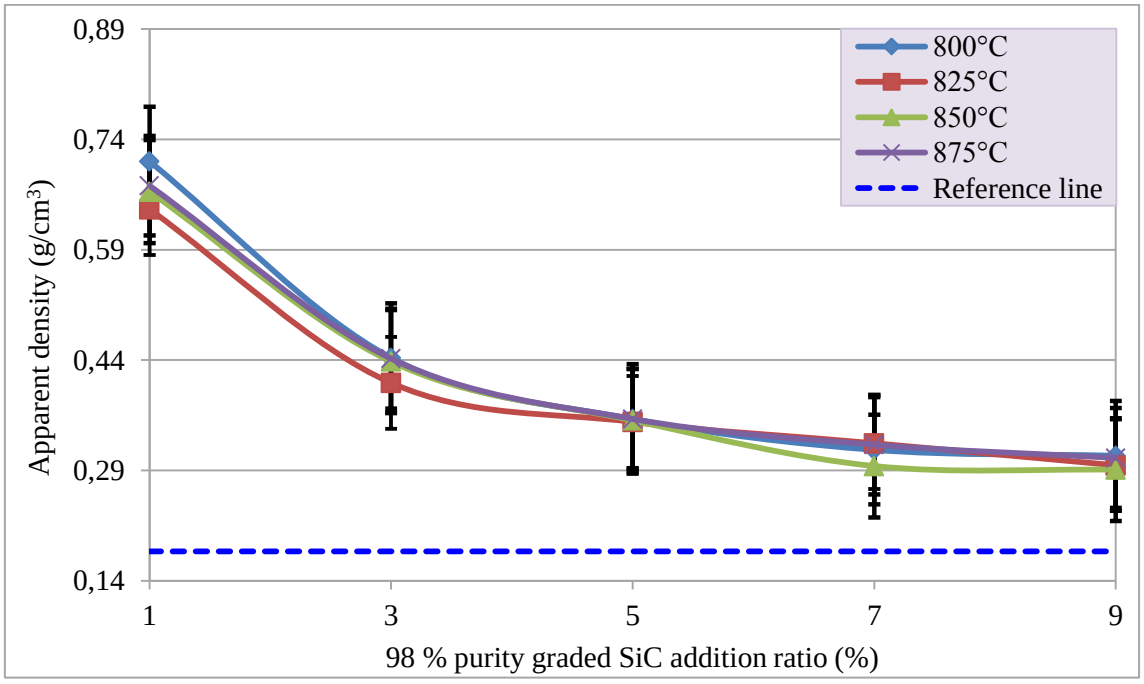


Figure 3.34. Apparent density variation of 98% grade SiC addition ratio of foam glass samples.

When all foam glass samples are compared according to density values, satisfactory low density values were achieved from 90 % purity graded SiC foam glasses. Other samples produced with using 95 and 98 % purity graded SiC had high density according to the standard values. 90 % purity graded SiC provided better foaming ability and convenience in comparison with 95 and 98 % purity graded SiC foaming agents at the same composition and sintering temperatures.

Also, the lowest density was obtained as 0.164 g/cm^3 from the sample (90-5-C) produced with using 90 % purity graded SiC with a ratio of 5 wt. % and sintered at 850°C. Moreover, the sample 90-5-C, some other foam glass samples (90-3-B, 90-5-B, 90-7-B, 90-9-B, 90-7-C and 90-9-C) with using 90 % purity graded SiC (with a ratio of 3, 5, 7 and 9 wt. % and sintered at 825 and 850°C) could provide the desired low density below 0.180 g/cm^3 as illustrated in the Table 3.12. These results meet the required standard values (<0.18 g/cm^3) and provide seven different alternative lightweight foam glass materials which can be selected considering to their other desired properties. However, the density values of the 95 and 98 % purity graded SiC foam glasses were found higher; consequently these high density would negatively affect the insulation properties.

3.3.5. Short term water absorption by partial immersion

Related standards suggested that short term water absorption by partial immersion values should be measured in the SI units of kg/m^2. Thus, measured values were transformed from g/mm^2 to kg/m^2. Short term water absorption test illustrates the water penetration to the product by exposing to rainy condition for 24 hours. In general, the penetrating water content (in terms of kg) to the unit area (in terms of m^2) should be lower than 0.5 kg/m^2 according to the TS EN 13167:2013-04 [24].

Short term water absorption test was applied on some selected samples to observe the effect of foaming agent addition ratio (1, 5 and 9 wt.), SiC foaming agent purity grade (90, 95 and 98%) and sintering temperature (at 800 and 850°C). Results were given in the Table 3.13.

Table 3.13. Short term water absorption values (kg/m^2) of selected samples.

SiC purity grade (%)	Temperature (°C)	SiC addition ratio (wt. %)		
		1	**5**	**9**
90	**800**	3.50	1.96	4.02
	850	9.08	7.92	4.87
95	**800**	3.33	2.96	2.71
	850	4.78	2.81	2.77
98	**800**	0.64	2.28	2.72
	850	3.70	5.96	9.67

However, no distinct correlation was found between water absorption and foaming agent addition ratio; or between water absorption and foaming agent purity grade. It can be seen from the graphs which are given in the Figure 3.35 and Figure 3.36.

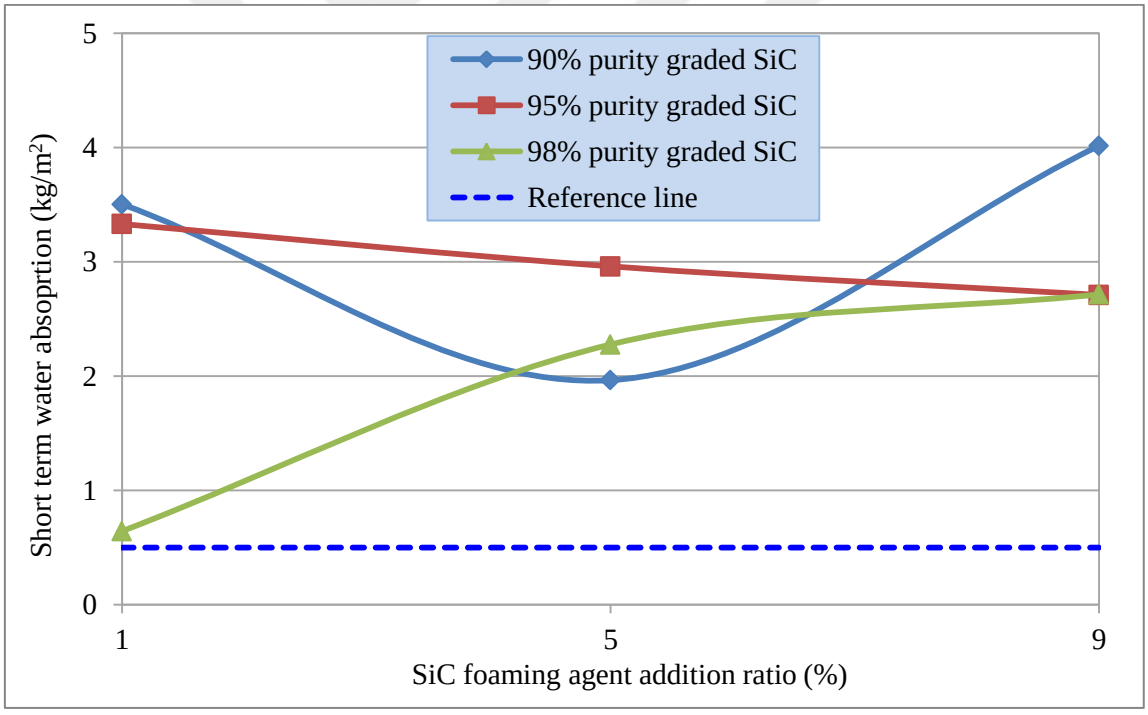


Figure 3.35. Short term water absorption by partial immersion graph of the selected foam glass samples sintered at 800°C.

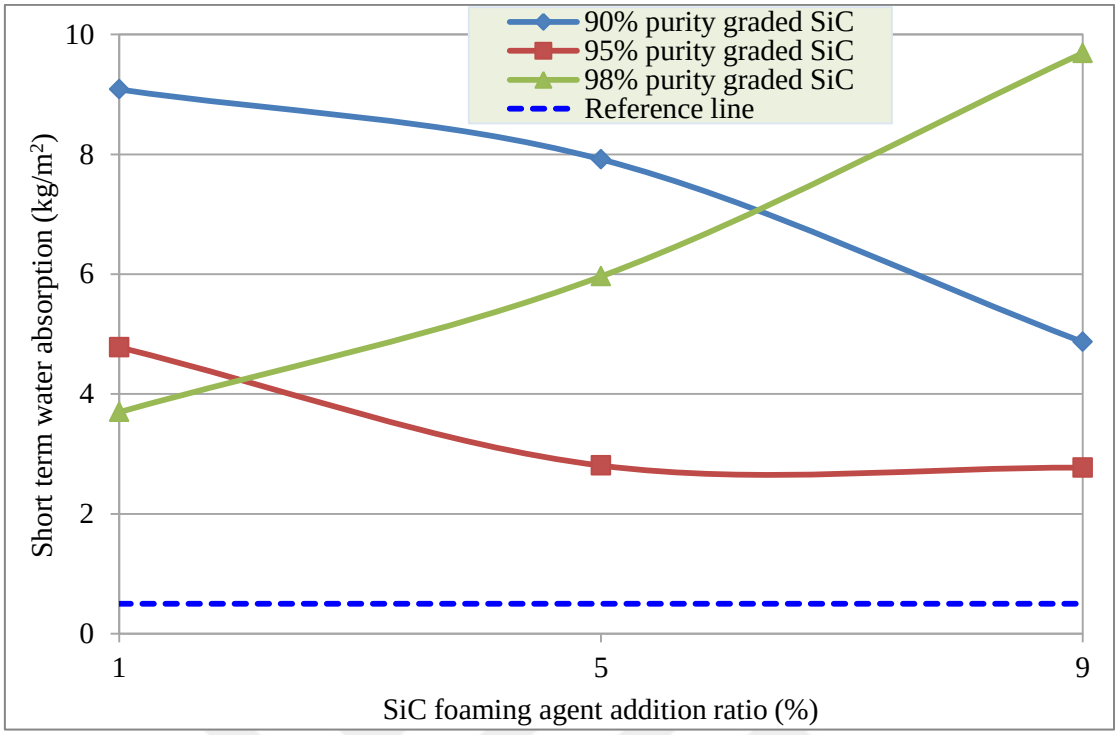


Figure 3.36. Short term water absorption graph of the selected foam glass samples sintered at 850°C.

From the Table 3.13 and Figure 3.35-36, the lowest water absorption value was achieved from the 98 % purity graded SiC with a ratio of 1 wt. % and sintered at 800°C. This result can be anticipated because this sample (98-1-C) was produced with the highest purity graded SiC with lowest wt. % addition and sintered at the lowest temperature, thus it was nearly a sintered glass material with having very few porosity. Since the glass does not absorb the water, the sample is far away from a foam glass material by providing the lowest short term water absorption value with 0.64 kg/m^2.

Also, no relation was found between water absorption and density values as reported in a study [67] conducted by Spiridonov et al. (2003). But temperature increasing gave sensible results to understand short term water absorption properties of foam glass. Additionally to Table 3.13, effect of sintering temperature on short term water absorption values were given in Table 3.14. It can be seen from the Table 3.13 and Table 3.14, water absorption values increased by rising sintering temperature.

Table 3.14. Short term water absorption values (kg/m^2) according to sintering temperatures.

SiC purity grade (%)	Temperature (°C)	SiC addition ratio (5 wt. %)
90	800	1.96
	825	2.94
	850	7.92
	875	8.33

The lowest water absorption values of 90 % purity graded SiC foam glass was observed with addition of 5 wt. % SiC at 800°C; moreover, this value rose at 875°C. Through temperature increase, initially expanded and foamed structure began to collapse and melting. Also, the cells began to enlarge and interconnect with each other. Larger cells tend to absorb higher amount of water, thus as given in Table 3.13 and 3.14 samples revealed higher water absorption values at higher sintering temperature.

In addition, significant rising of water absorption was observed which could be caused with the same reason discussed above. At 800°C, the pressed samples could not be sintered enough for transforming into foam glass material. The samples sintered at lower temperatures may have more amorphous phase rather than other samples sintered at higher temperatures. Thus, the samples sintered at lower temperature revealed glass phase due to the fact that the temperature is not enough to start any foaming reaction and behave like glass without absorption water. The temperature effect on short term water absorption was graphically illustrated in Figure 3.37.

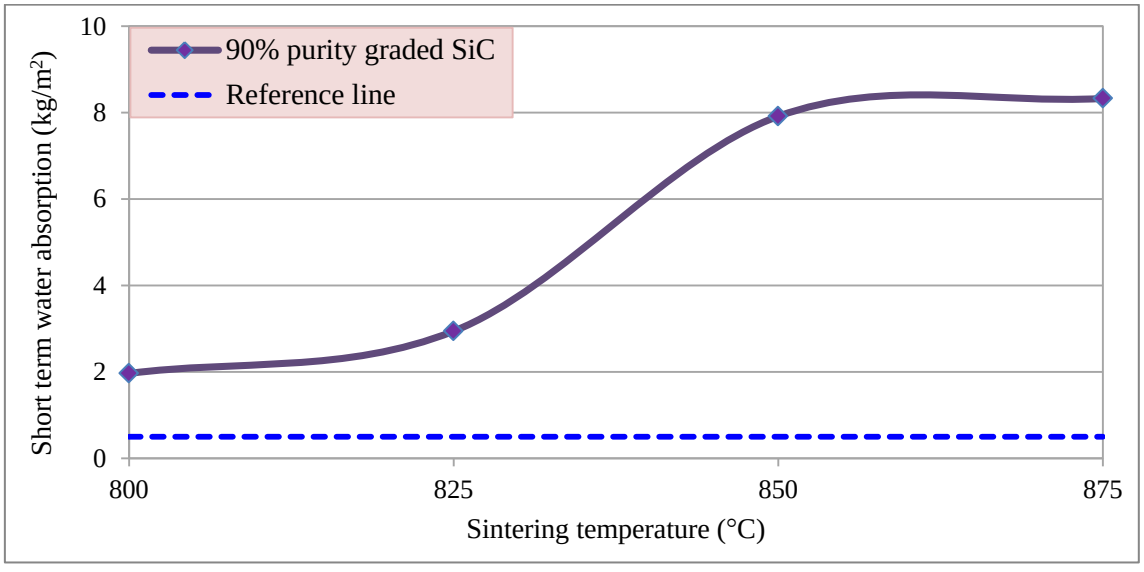


Figure 3.37. Short term water absorption by partial immersion graph of the foam glass samples with using 90 % purity graded SiC with a ratio of 5 wt. % at 800, 825, 850 and 875°C.

3.3.6. Long term water absorption by total immersion

It is indicated in the standard TS EN 13167:2013-04 that long term water absorption by total immersion values of foam glass should be very low (<0.2 %). However, long term water absorption values of the selected samples were found higher than related standard value. The results of selected samples were presented in Table 3.15.

Table 3.15. Long term water absorption values (%) of selected foam glass samples.

SiC purity grade (%)	Temperature (°C)	SiC addition ratio (wt. %)		
		1	5	9
90	800	35.28	18.13	31.73
	850	65.72	56.71	35.31
95	800	34.05	28.99	26.81
	850	37.35	23.31	26.16
98	800	21.19	38.40	27.45
	850	32.48	43.71	69.56

Similar to the short term water absorption test results, no correlation was found between long term water absorption values and foaming agent addition ratio or between long term water absorption values and foaming agent purity grade as seen from the graphs illustrated in Figure 3.38 and Figure 3.39.

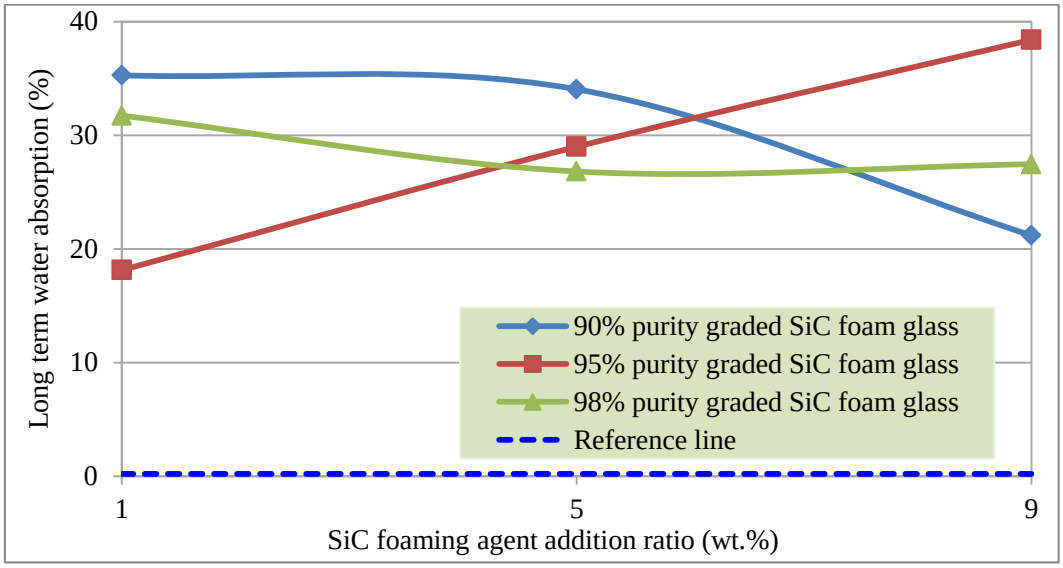


Figure 3.38. Long term water absorption by total immersion (%) graph of the selected foam glass samples sintered at 800°C.

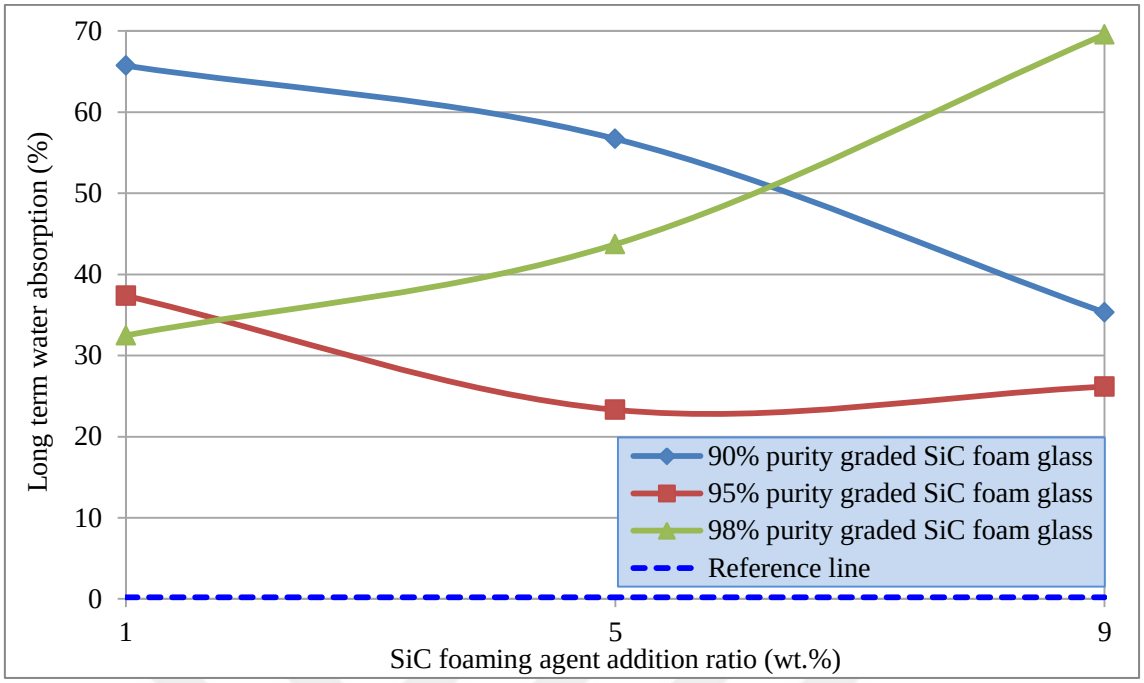


Figure 3.39. Long term water absorption by total immersion graph of the selected foam glass samples sintered at 850°C.

Similar to the short term water absorption results, the lowest long term water absorption by total immersion was measured from the sample produced with 1 wt. % addition of 98 % purity graded SiC and sintered at 800°C. This sample (98-1-C) provided the lowest volumetric expansion and lowest porosity among all foam glass samples; moreover, it was nearly a sintered glass material with containing very few cells as mentioned in the previous section. Thus, this sample is far away from foam glass material requirements by having the lowest long term water absorption value with 20.19 %. Temperature effect was more specifically seen from the Table 3.16 and Figure 3.40.

Table 3.16. Long term water absorption values (%) according to sintering temperature.

SiC purity grade (%)	Temperature (°C)	SiC addition ratio (5 wt. %)
90	800	18.13
	825	24.05
	850	56.71
	875	57.09

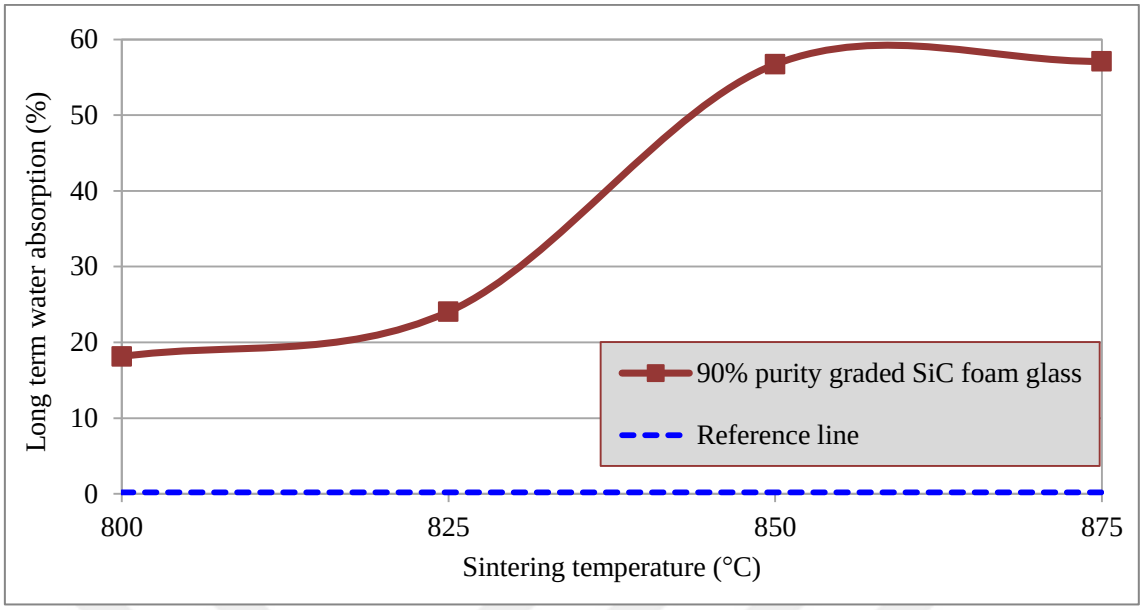


Figure 3.40. Long term water absorption by total immersion graph of the foam glass samples with 90 % purity graded SiC with a ratio of 5 wt. % at 800, 825, 850 and 875°C.

After exposing of water by total immersion for 28 days, it was measured that the foam glass samples produced with addition of 5 wt. % SiC (90 % purity graded) and sintered at 800°C displayed the lowest long term water absorption value. By increasing of the sintering temperature to 875°C, water absorption values were also increased. Similar explanations which were made with short term water absorption can also be valid. Temperature rising enhanced the cell sizes of foam glass, and then initially smaller cells tended to interconnect with each other and finally enlarged and locally melt. Coarser cells can absorb the water easily and water can penetrate and go through in the structure which has high amount of large cells resulting in higher water absorption values.

Moreover, 800°C temperature may be insufficient to complete foaming reactions because of the high melting point SiC used as foaming agent. At this relatively low temperature, satisfactory sintering couldn't be achieved for transforming the mixture to foam glass. Thus, amorphous phase from the glass may dominate the foam glass at lower sintering temperature; then, glassy phase revealed the lower water absorption rather than highly cellular glass foam. Finally, all the samples produced in this study absorbed high amount of water. This is due to the fact that SiC used as reducing agent (foaming agent), hence final products had crystalline phases after foaming reactions. During sintering amorphous glass is transformed into partially crystalline foam glass materials which lead to high water absorption in the samples.

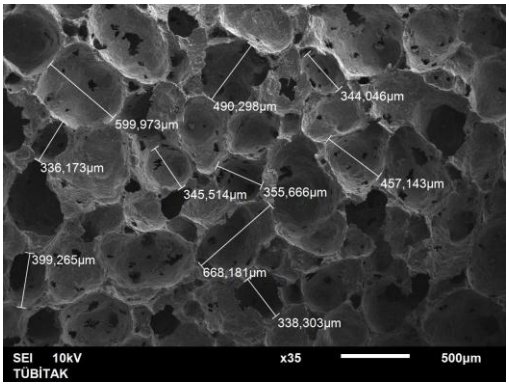
3.3.7. Microstructural evaluation (SEM)

According to microstructural examination conducted by SEM, cellular structure was observed at all foam glass samples. These microstructures were illustrated in Figure 3.41. Increasing of foaming agent addition ratio from 1 wt. % to 9 wt. % caused a remarkable pore size enlargement. Each purity graded SiC foaming agents (90, 95 and 98 % purity) addition resulted in increasing cell size particularly with higher addition ratios. This pore size enlargement was more significant at the samples produced with 90 % purity graded SiC as illustrated in Figure 3.41(a-b). Because of the foaming reaction take place readily at the 90% purity graded SiC foam glasses, gas bubbling started earlier and enlargement of pores occurred more efficiently than others during the same reaction time. In other words, there was more time for evolving and expansion of gases in 90 % purity graded SiC foam glasses; on the other hand, because of the highest purity which is 98 % purity graded SiC hardly reacted and caused evolving of smaller bubbles. Thus, generated bubbles were still smaller than other purity graded SiC foam glass even though with addition of high amount of 98 % purity graded SiC.

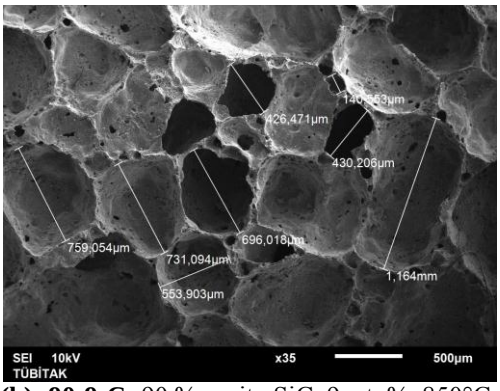
Sintering temperature effect can be seen from the Figure 3.41(g-h). At 800°C, expansion ratio was lower than the foam glass sintered at 875°C. Thus, the cells were smaller and had more homogeneous size distribution, nevertheless, with increasing temperature, cells began to expand and there was a little inhomogeneous larger cell size distribution occurs.

Sintering at higher temperatures like 875°C, microstructure degradation starts and walls or struts between the cells began to be destroyed or teared. This behavior can be attributed to tearing of the walls which may result from the pressure build-up inside the cells or pores with rising temperature.

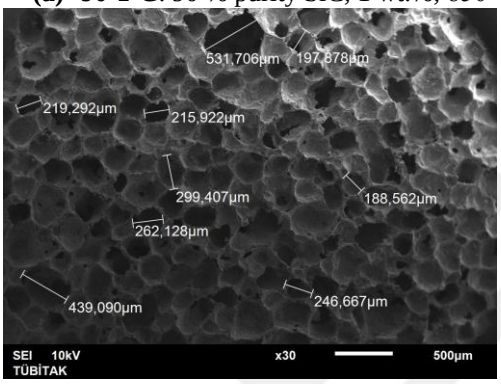
It can be concluded that increase in sintering temperature cause mechanical strength reduction. Both of the generating voids within the larger cells and tearing walls are caused by temperature increment, and these are one of the important reasons of strength reduction of foam glass. These consequences are compatible with compressive strength results, because it was also showed that compressive strength values decreased with increasing temperature as presented in Table 3.11.



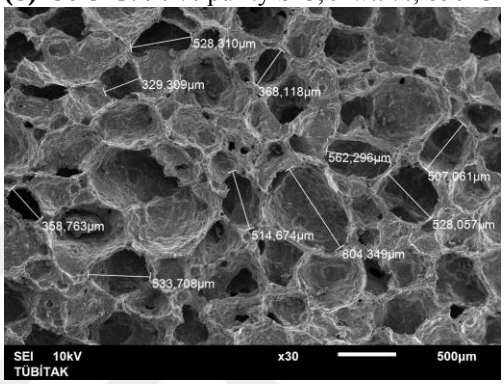
(a) 90-1-C: 90 % purity SiC, 1 wt.%, 850°C



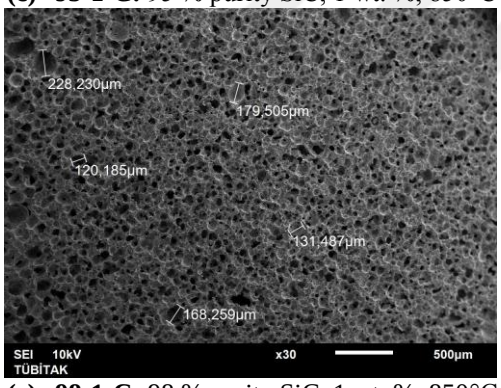
(b) 90-9-C: 90 % purity SiC, 9 wt. %, 850°C



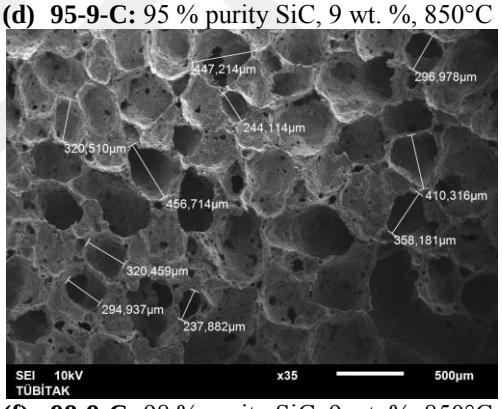
(c) 95-1-C: 95 % purity SiC, 1 wt. %, 850°C



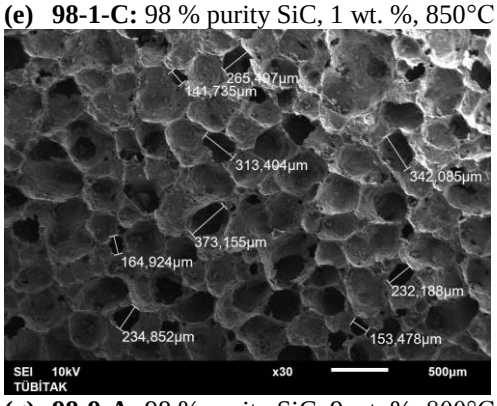
(d) 95-9-C: 95 % purity SiC, 9 wt. %, 850°C



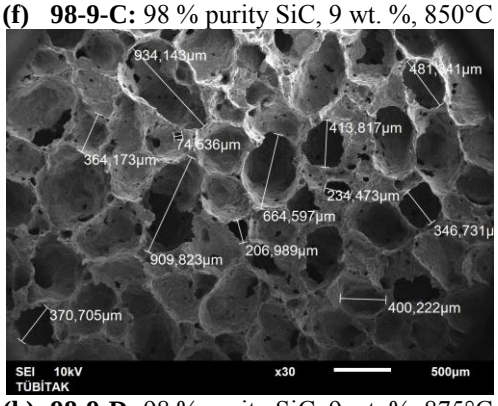
(e) 98-1-C: 98 % purity SiC, 1 wt. %, 850°C



(f) 98-9-C: 98 % purity SiC, 9 wt. %, 850°C



(g) 98-9-A: 98 % purity SiC, 9 wt. %, 800°C



(h) 98-9-D: 98 % purity SiC, 9 wt. %, 875°C

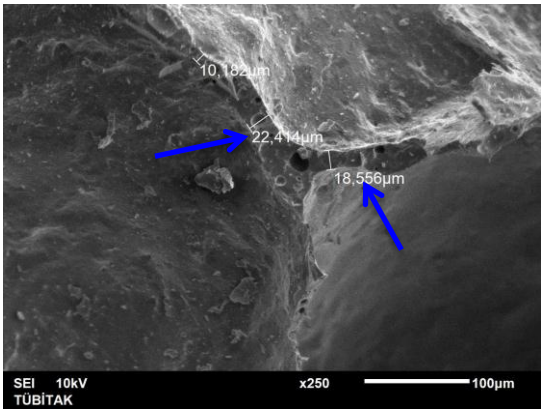
Figure 3.41. Porous structures of the selected foam glass samples observed by SEM.

Increasing of foaming agent purity grade positively affects the homogeneity of cell size distribution. It can be easily observed from the Figure 3.41(a), (c) and (e). SiC purity

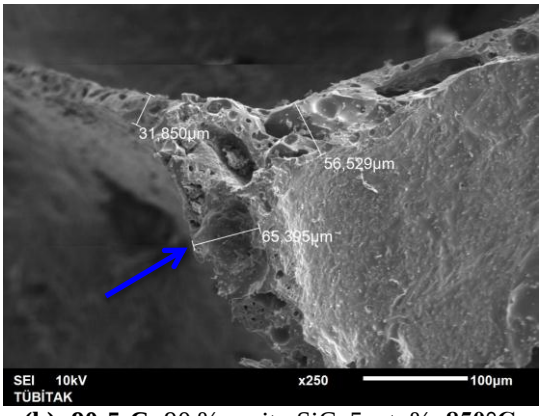
grade is another important effective parameter on compressive strength. Higher purity grades of SiC leads to smaller cell size which resulted in higher strength with respect to larger cells. It can be seen from the Figure 3.41(e), the smallest pore size was achieved from the highest purity graded SiC with lowest addition. Moreover, the largest pore size was obtained from the lowest purity graded SiC with highest addition ratio (Figure 3.41(b)). These results are also compatible with compressive strength values. In other words, foaming affinity can be observed from the pore size enlargements which reduce the strength in connection with the pore size.

An interesting correlation found among the strut (cell wall) thickness and purity grade of SiC, SiC addition ratio and sintering temperature of foam glass. Firstly, there is a correlation between the strut thickness of foam glass and with mechanical strength. It was observed that larger void size or cell size causes an increase in strut thickness. Also, increasing of strut thickness results in mechanical strength reduction due to those larger voids framed by glass wall. Strut images and thicknesses of some selected samples were illustrated in Figure 3.42.

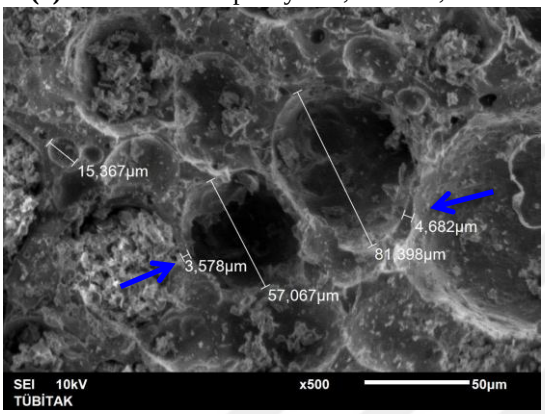
From the Figure 3.42 (a) and (b), strut thickness increased with increasing temperature from 800°C to 850°C. It can be explained that rising temperature enhanced the foaming, reactions and voids enlargement caused to thickening of struts. The compressive strength results showed that the sample 90-5-A (sintered at 800°C) had 2.18 MPa, while 90-5-C (sintered at 850°C) provided 1.50 MPa. Thus, increasing temperature caused strut thickening with decreasing strength. The thinnest strut (4 μm) was achieved from the sample 98-1-A which has the highest purity grade SiC with lowest addition ratio and sintered at lowest temperature. Also, this sample revealed the highest compressive strength with 48.98 MPa, thus the strut thickness effect was proved by this result. However, cell size was increased by increasing of addition ratio from 1 wt. % to 9 wt. % and this resulted in strut thickness increasing (22 μm), also (Figure 3.42 (d)). Increasing of strut thickness from 4 μm to 22 μm, caused a significant strength reduction from 48.98 MPa to 7.04 MPa.



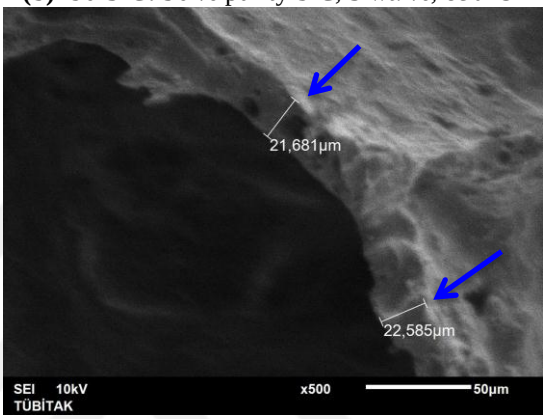
(a) 90-5-A: 90 % purity SiC, 5 wt. %, **800°C**



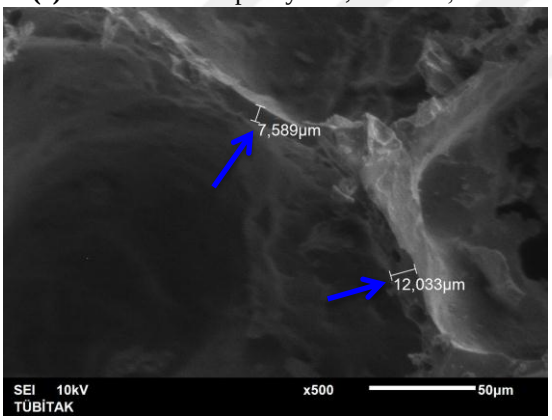
(b) 90-5-C: 90 % purity SiC, 5 wt. %, **850°C**



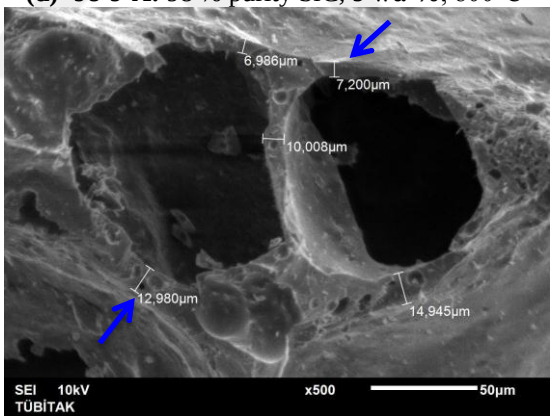
(c) 98-1-A: 98 % purity SiC, **1 wt. %**, 800°C



(d) 98-9-A: 98 % purity SiC, **9 wt. %**, 800°C



(e) 98-5-A: 98 % purity SiC, 5 wt. %, 800°C



(f) 95-5-A: 95 % purity SiC, 5 wt. %, 800°C

Figure 3.42. Strut (cell wall) views and thickness of selected foam glass samples.

In addition, foaming agent purity grade affected strut thickness and correspondingly strength values. 90 % purity graded SiC provided the thickest strut according to 95 and 98 % purity graded SiC foaming agents. With increasing purity degree, strut thickness declined from 10-22 µm, 6-14 µm and 7-12 µm (Figure 3.42(a), (f) and (e), respectively). These strut thickness reductions led compressive strength increase and provided the following values: 2.18 MPa, 3.83 MPa and 10.10 MPa for Figure 3.42(a), (f) and (e), respectively.

3.3.8. Thermal conductivity

Thermal conductivity is one of the most important properties of foam glass. To maintain a sufficient thermal insulation, this value should be lower <0.065 W/mK according to TS 825 standard [7]. Also, thermal conductivity value for foam glass material was specified to be 0.045 – 0.060 W/mK at the TS EN 13167:2013-04 standard [24].

Thermal conductivity analyses were performed for some selective foam glass samples to see the effect of sintering temperature, foaming agent addition ratio and foaming agent purity grade. Within this scope, eight samples were tested and results were shown in Table 3.17.

According to Table 3.17, almost each one of these analysed samples met the standart thermal conductivity value below 0.065 W/mK, thus each one of them can be evaluated as an insulation material based on the "Thermal insulation requirements for buildings – TS 825" [7]. To distinguish the sintering temperature effect on thermal conductivity, a graph was shown in Figure 3.43.

Table 3.17. Thermal conductivity (W/mK) values of selected foam glass samples.

Sample Code	SiC purity grade (%)	SiC addition ratio (wt. %)	Sintering temperature (°C)	Thermal conductivity, λ (W/mK)
90-5-A	90	5	800	0.048
90-5-B	90	5	825	0.045
90-5-C	90	5	850	0.046
90-5-D	90	5	875	0.047
90-1-C	90	1	850	0.076
90-9-C	90	9	850	0.052
95-5-C	95	5	850	0.057
98-5-C	98	5	850	0.065

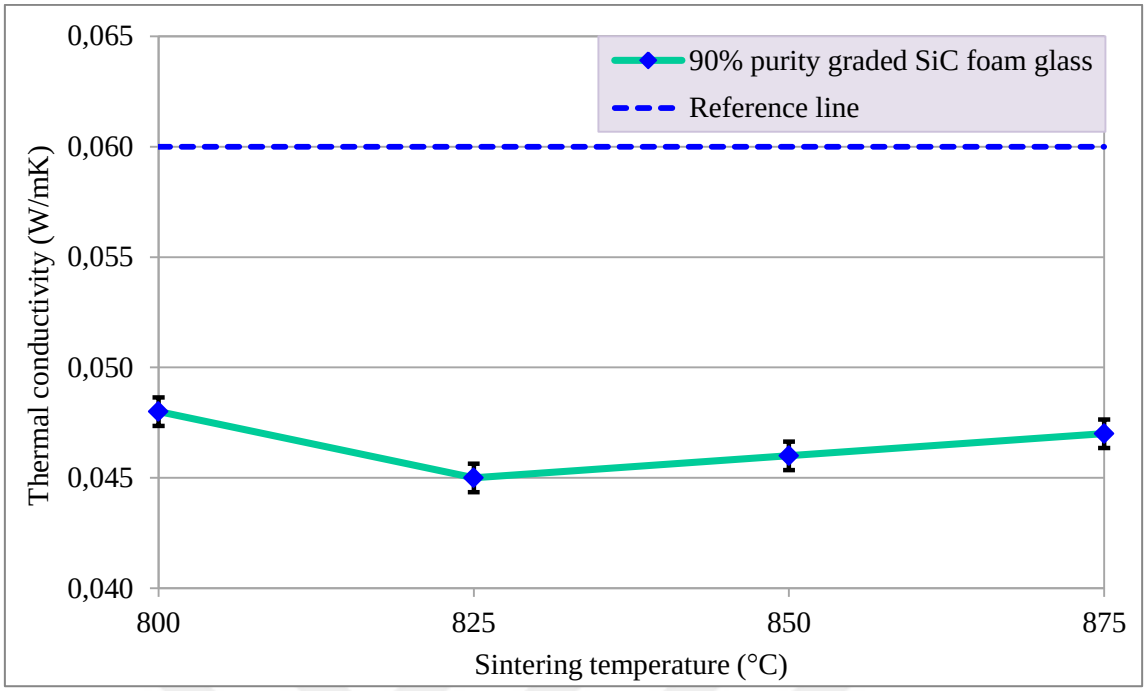


Figure 3.43. Variation of thermal conductivity with sintering temperature for foam glass samples containing 5 wt. % SiC foaming agent (90 % purity graded).

It was seen from the Figure 3.43 that the best results were obtained from the middle zone of the temperatures like 825 and 850°C. When temperature decreased to 800°C or increased to 875°C, λ value began to increase. These results highly depend on the microstructure and density of the foam glasses. Firstly, it was indicated that the most proper volume expansion, in other words gas bubbling and porosity, was achieved at 825 - 850°C. At lower temperature like 800°C, there wasn't enough porosity and cells were too small because the foaming reaction could not be completed yet. At higher temperature like 875°C, however, some distortion and collapsing of cells began due to starting of melting. Pores and cells generate interconnection with each other and closed cells got started to disappear because of these distortion at high temperature. These undesired pore structures negatively affected the thermal conductivity of samples. On the other hand, this behavior can be attributed to density increasing effect on the thermal conductivity. As it was mentioned before in Table 3.12, sintering of foam glass at low temperature (800°C) and high temperature (875°C) caused an increase on density. Moreover, the thermal conductivity results of the samples are dependent on their apparent density results. For these samples given in the Figure 3.43, thermal conductivity values decreased with decreasing density; similarly, higher density led an

increase in thermal conductivity. One of the important reasons of density increment is sintering temperature because it is the only variable when other parameters (purity grade and addition ratio etc.) were constant. Thus, it can also be said that sintering temperature is quite effective on thermal conductivity.

Another important factor to influence thermal conductivity values is SiC foaming agent addition ratio (wt. %). Figure 3.44 illustrates the changing of λ value based on the 90 % purity graded SiC foaming agent addition ratio.

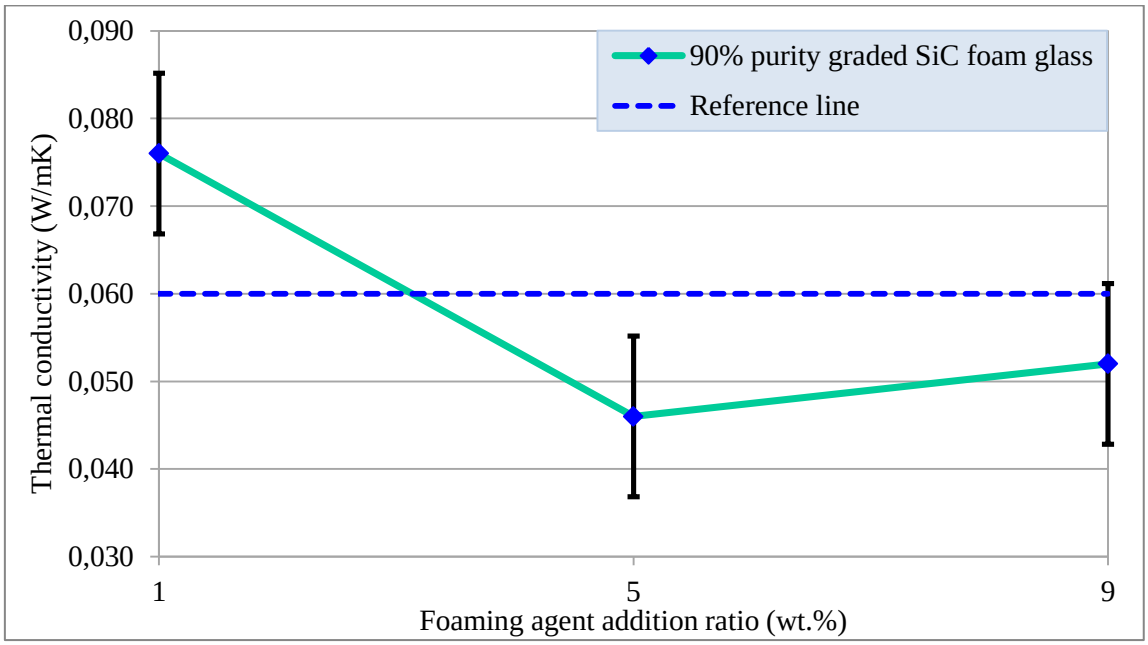


Figure 3.44. Variation of thermal conductivity with foaming agent addition ratio for foam glass samples containing 1, 5 and 9 wt. % SiC foaming agent (90 % purity graded) at 850°C.

As a result of porous structure, density were also influenced by foaming agent addition ratio, thermal conductivity values were also changed according to increasing of foaming agent addition. From the Figure 3.44, the highest and lowest result was obtained at 1 wt. % SiC (90 % purity) addition suprisingly. It was specified at previous sections, 1 wt. % SiC addition could not provide the sufficient porosity and density because of high glass content. However, better volume expansion, better proper porous structure and lowest density were achieved from the sample 90-5-C which was produced with 5 wt. % SiC (90 % purity) and sintered at 850°C. Thus, the lower density and lower porosity provide lower thermal conductivity value. As with density results of these samples, 9 wt. % SiC addition caused an increase on thermal conductivity value. Similarly, a correlation between the density and thermal conductivity was shown by these results.

Interestingly, it was observed that another correlation was existed between foaming agent purity grades and thermal conductivity. The lowest thermal conductivity value was achieved from the 90 % purity graded SiC foam glass samples; beside, λ value inceased with the increasing of SiC purity grade as illustrated in the Figure 3.45.

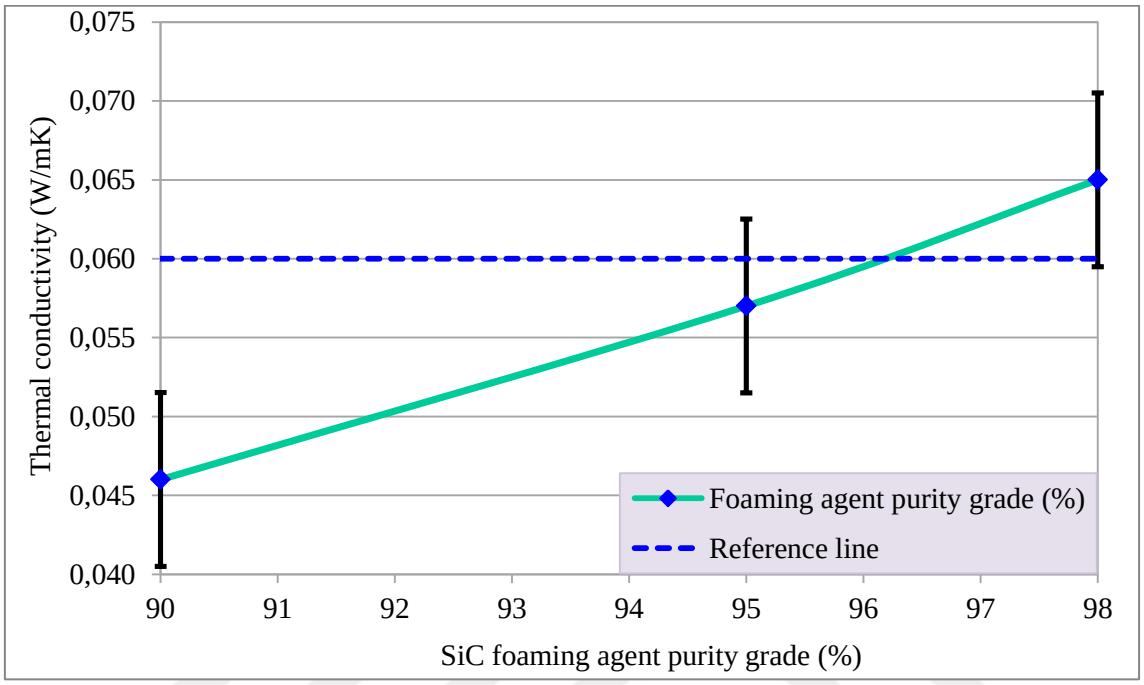


Figure 3.45. Variation of thermal conductivity with foaming agent purity for foam glass samples containing 90, 95 and 98 % SiC (5 wt. %) sintered at 850°C.

Variation of SiC purity dominates the changing of thermal conductivity value for foam glass. Density increasing through the 98 % purity graded SiC foam glass, thermal conductivity increased also. This may be attributed to the high thermal conductivity feature of SiC which contributes to the overall thermal conductivity as the SiC purity raises. On the other side, porous structure may influence the results. The sample 90-5-C, had the bigger closed cells and relatively homogeneous porous structure therefore it can be said that 90 % purity graded SiC could react easier rather than 95 and 98 % purity graded SiC foaming agents.

3.3.9. Total porosity

Porosity is a distinctive property for foam glass materials. There isn't a limit value for desired porosity level in related standards. However, it is expected that porosity of foam glass should be higher as possible. Similar to previous sections, various important properties like density, mechanical strength and thermal conductivity strongly depend on porous structure of foam glass. Thus, porosity can change the foam glass features.

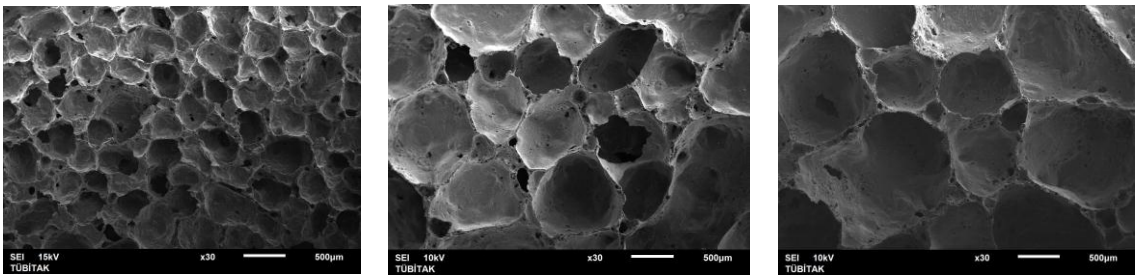
Existence of high porosity results in low density, low thermal conductivity and lower mechanical strength. In addition, low porosity leads to density increasing and mechanically strength materials but weak thermal insulation. To obtain these characteristics, total porosity (open and closed together) values of some selected foam glass samples were measured and analyzed based on the foaming agent addition ratio, sintering temperature and foaming agent purity grade. Results are presented in Table 3.18.

Table 3.18. Total porosity values (%) of selected foam glass samples.

Sample name	SiC purity grade (%)	SiC addition ratio (wt. %)	Sintering temperature (°C)	Total porosity (%)
90-1-C	90	1	850	15.45
90-5-C	90	5	850	76.77
90-9-C	90	9	850	83.61
90-5-D	90	5	875	61.82
95-5-C	95	5	850	65.34
98-5-C	98	5	850	51.50

To understand the foaming agent addition ratio effect on porosity, the samples produced with 1, 5 and 9 wt. % SiC (90 % purity) addition were investigated. It was also seen from the SEM analysis (Figure 3.46), increasing of foaming agent addition ratio dominantly enlarged the pore sizes and caused larger voids within the cells. But high amount of SiC resulted in heterogeneous pore size distribution which contains smaller cells on the walls of larger cells. With high amount of SiC addition, foaming reaction was accelerated and initially smaller cells continued to grow and generate larger pores which were also occur from combining with each other. Figure 3.47 shows the total porosity (%) values of the samples with using 1, 5 and 9 wt. % SiC (90 % purity), also it

can be seen the microstructures of these samples from the Figure 3.46.



(a) 1 wt. % SiC (90 % purity) **(b) 5 wt. %** SiC (90 % purity) **(c) 9 wt. %** SiC (90 % purity)

Figure 3.46. Pore sizes and structures of the samples with using **(a)** 1 wt. %, **(b)** 5 wt. % and **(c)** 9 wt. % SiC (90 % purity graded) and sintered at 850°C.

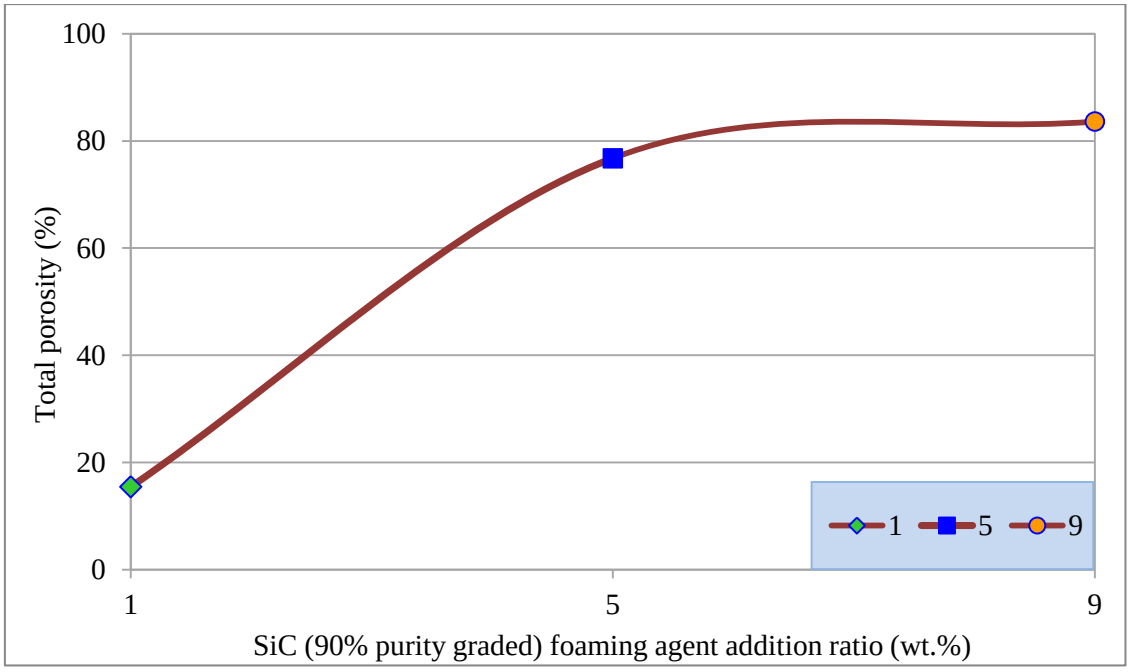


Figure 3.47. Total porosity variations of foam glass samples with 1, 5 and 9 wt. % SiC (90 % purity graded) foaming agent and sintered at 850°C.

Porosity results are comparable with the density and compressive strength results. The increasing of porosity with addition of higher amount of SiC, decreased the density and compressive strength which were given in the Table 3.11 and 3.12. The sample 90-1-C has 15.45 % total porosity, 0.251 g/cm^3 apparent density and 4.23 MPa compressive strength values; on the other hand, the sample 90-9-C which contains 9 wt. % SiC has 83.61 % total porosity, 0.174 g/cm^3 apparent density and 1.23 MPa compressive strength. In addition, the sample 90-1-C which has lower porosity and higher density obtained 0.076 W/mK thermal conductivity value; while the sample 90-9-C which has higher porosity and lower density provided 0.052 W/mK thermal conductivity value.

Thus, it was proved that porosity is extremely effective on density, mechanical strength and thermal conductivity properties.

Another factor to affect the porosity is temperature. 76.77 % porosity was achieved from the sample sintered at 850°C whereas the same sample was sintered at 875°C but lower porosity obtained as 61.82 %. This result can be attributed relatively higher volume expansion of the sample 90-5-C (sintered at 850°C) than the same sample 90-5-D sintered at 875°C. With increasing sintering temperature, partially melting started and tearing of walls began which led to softening and distortion of foam glass. The highest expansion and porosity were achieved for samples fired at 850°C, but distortion and collapsing started above this temperature. Similarly, density and compressive strength values increased depending on lower porosity. Also, there was slight increase on thermal conductivity with decreasing of porosity. The temperature effect on porosity was given in Figure 3.48.

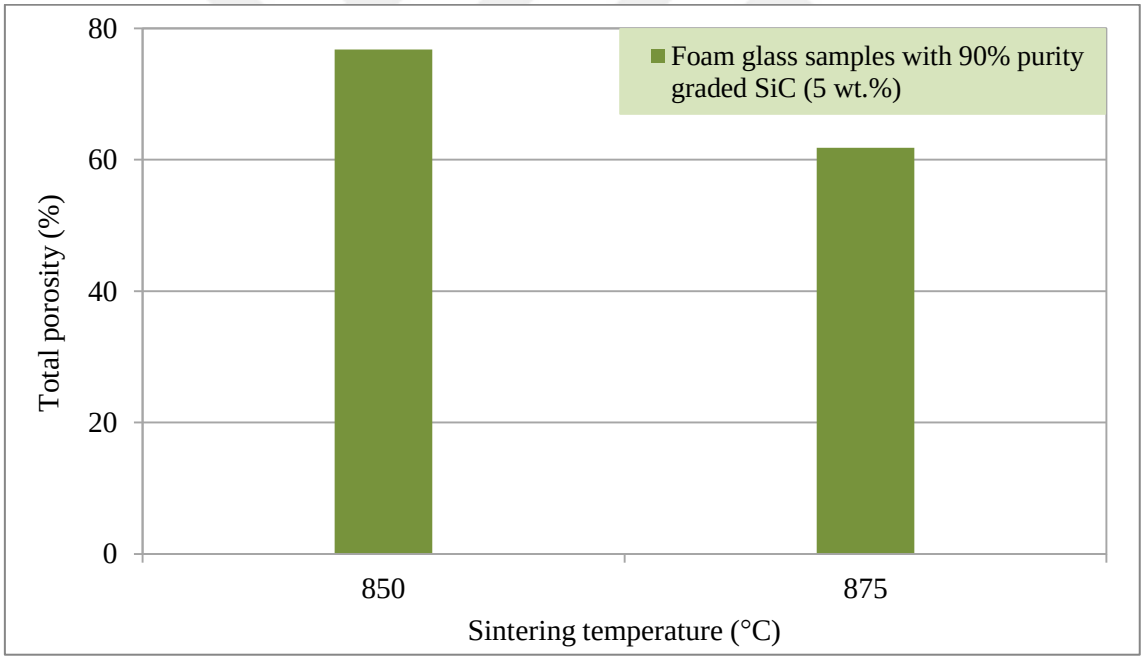


Figure 3.48. Total porosity graph of foam glass samples produced with using 5 wt. % SiC (90 % purity graded) foaming agent and sintered at 850 and 875°C.

Finally, it was seen that SiC purity grade affected also total porosity of foam glass just effecting like other features. Increasing of SiC purity grade made difficult to foam the glass, total porosity values and pore size distribution decreased by increasing of foaming agent purity grade. As it mentioned in previous sections, 90 % purity grade SiC

enhanced the foaming process and highly porous structures could be achieved with high amount of larger cells. However, the foaming process hardly proceeds with increasing of foaming agent purity. When a comparison was made among 90, 95 and 98 % purity grade SiC; the highest volume expansion and larger cells were obtained at 90 % purity graded SiC foam glass sample resulted in the highest porosity. Meanwhile, total porosity value declined with increasing foaming agent purity and cell size distribution as illustrated in Figure 3.49 and Figure 3.50.

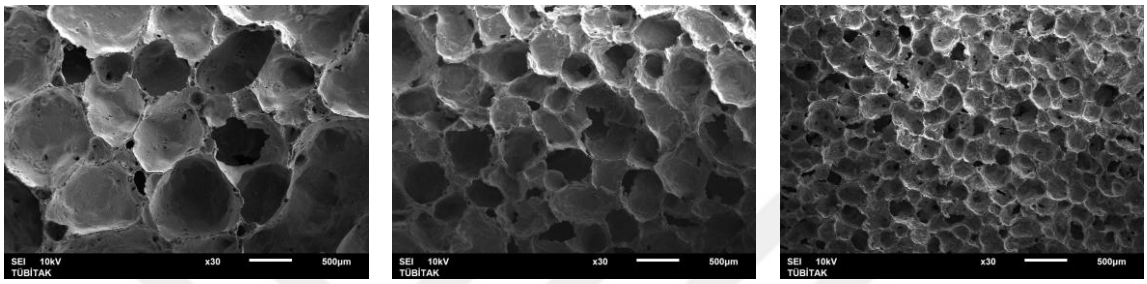


Figure 3.49. Pore structures of the samples with **(a)** 90 % purity, **(b)** 95 % purity and **(c)** 98 % purity graded SiC foaming agent with a ratio of 5 wt. % and sintered at 850°C.

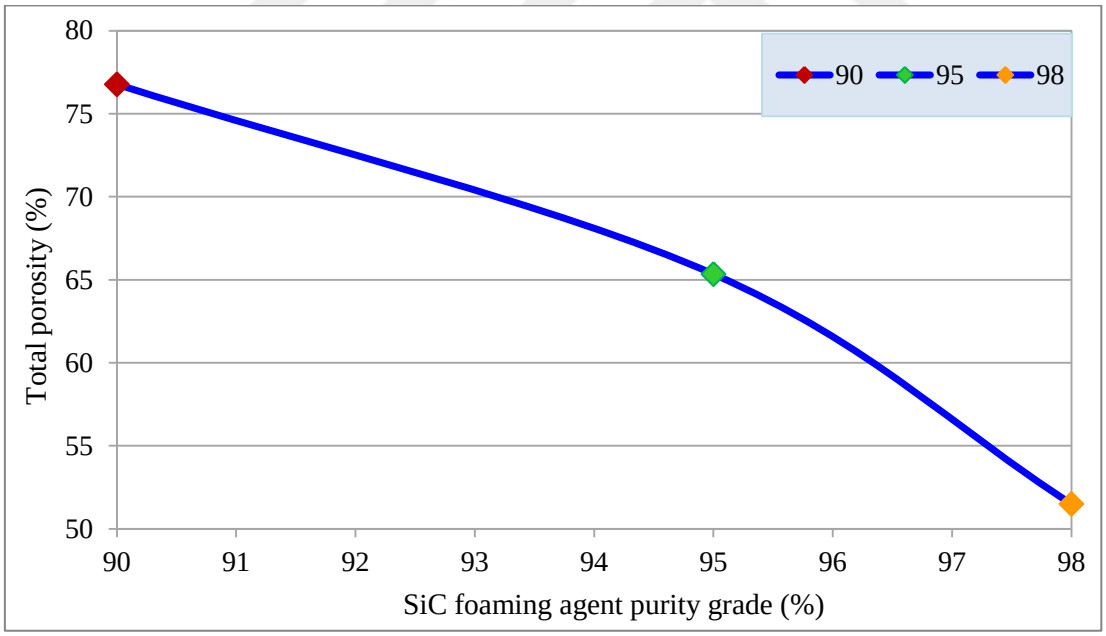


Figure 3.50. Total porosity variations of foam glass samples with 90, 95 and 98 % purity graded SiC foaming agent (5 wt. %) and sintered at 850°C.

The porosity results of different purity graded SiC foam glass were consistent with density, compressive strength and thermal conductivity values. By increasing of purity grade of SiC, density, compressive strength and thermal conductivity values increased which lead to decrease in total porosity. The highest porosity was achieved from the 90

% purity graded SiC foam glass, and this higher porosity provide lower density and lower thermal conductivity with mechanical strength reduction. Also, 98 % purity graded SiC foam glass caused decline in the porosity along with density, mechanical strength and thermal conductivity rising.

Curve-like expansion of foam glass samples was observed at the top of the body. It was indicated in the Foam Glass Market Survey Report written by Hurley [28], expansion of foam glass products start from the top surface. But the foam glass material provides insulation with inside, thus, the heat transfer during sintering may be prevented by the expanded top side which has porous structure leading to lower thermal conductivity. During heat treatment, the external side of foam glass is directly sintered and foaming take place, while foaming of the core of the body is not yet completed just because of this lower heat transfer and rapid external side insulation. Therefore, there is a possibility for overheating and collapsing of the cells at the top surface being intensively heated according to bottom part of the body [28]. This finding has a very good agreement with the present study results. Foaming and expansion of pores may begin from the external surface due to direction of heat transfer in the furnace. Because of the heat convection within the furnace, which supplies heat around and on top of the samples, and the samples was located on a stainless steel plate in the furnace. Hence, heat energy could not be transferred to the bottom side as effectively as in the top side of sample. Therefore, it was thought that the cells began to enlarge at the external side but the internal and bottom cells foaming generated later.

In 90 % purity graded SiC foam glass samples, the external cells slightly larger rather than the core and bottom cells revealing a distortion of sample during sintering.. This result was also obtained at 95 and 98 % purity graded SiC foam glass. However, the external cell size enlargement was more obvious in the 90 % purity graded SiC foam glass samples even they fired at the same sintering time. This behavior may be explained by 90 % purity graded SiC be able to give faster reaction due to its relatively higher impurity content rather than 95 and 98 % purity graded SiC agents. The cell size of 98 % purity graded SiC foam glass were smaller when compared with 90 and 95 98 % purity graded SiC because of slow rate of reactions due to inert nature of SiC at this temperature as shown in DTA/TG Analysis result (Figure 3.8) of 98 % purity grade SiC. Therefore no time was present to expand as like 90 % purity graded SiC foam

glass. The top surface and bottom surface cell distribution differences of both 90 and 98 % purity graded SiC foam glass was shown in Figure 3.51.

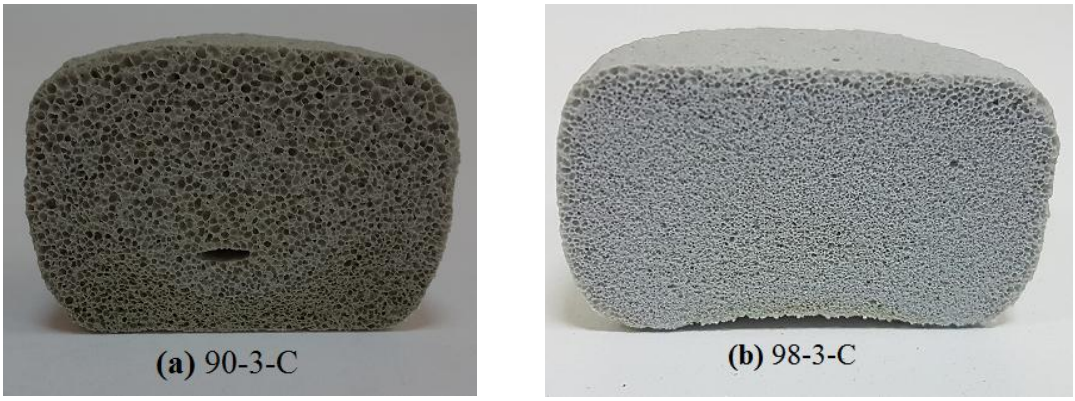


Figure 3.51. Top surface and bottom surface cell size distribution of foam glass samples with **(a)** 90 % and **(b)** 98 % purity graded SiC with a ratio of 3 wt. % and sintered at 850°C.

3.3.10. Coefficient of thermal expansion

In general, thermal expansion coefficient (K^{-1}) of foam glass products are specified between $8x10^{-6}$ K^{-1} to $10x10^{-6}$ K^{-1} at 23°C according to standard TS 13167:2013-04 [24], also the main commercial foam glass producer Pittsburgh Corning UK Ltd. [32] declared the value that it should be close to $9.0x10^{-6}$ K^{-1} at 25 to 300°C. To observe the thermal expansion behaviors of some selected foam glass samples and to compare them with commercial foam glass product, the tests were conducted at between 28-350°C.

Dimensional stability is one of the essential features of the foam glass materials. Therefore thermal expansion coefficient measurements were carried out which was provided insight about dimensional stability. Besides, these products are mainly used for heat insulation and as well as they are exposed to high and low temperatures. During this temperature changing, it should be dimensionally stable. The low thermal expansion coefficient value provides dimensional stability for foam glasses. Normally foam glass materials are dimensionally stable, because their thermal expansion coefficient values are relatively low. Table 3.19 shows the coefficient of thermal expansion values of selected samples; also variation of the thermal expansion of these samples with increasing temperature was depicted in Figure 3.52- Figure 3.54.

Table 3.19. Coefficient of thermal expansion (K^{-1}) values of selected foam glass samples.

Sample Code	SiC purity grade (%)	SiC addition ratio (wt. %)	Sintering temperature (°C)	Coefficient of thermal expansion (at 28-300°C) (K^{-1})
90-5-C	90	5	850	11.66×10^{-6}
95-5-C	95	5	850	11.84×10^{-6}
98-5-C	98	5	850	10.95×10^{-6}

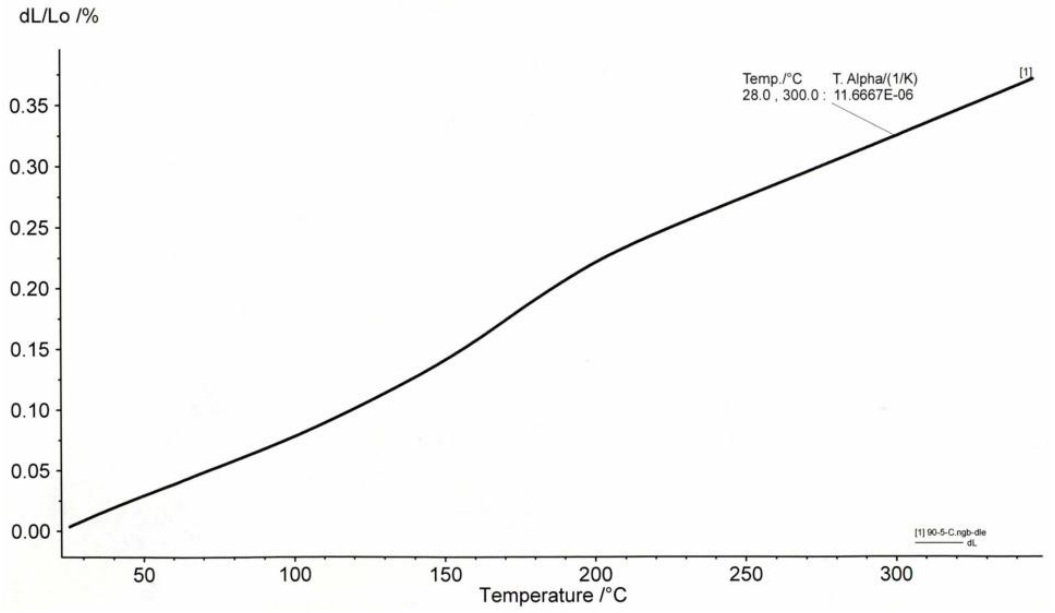


Figure 3.52. Coefficient of thermal expansion (K^{-1}) graph of the foam glass sample with 90 % purity graded SiC (5 wt. %) at 850°C.

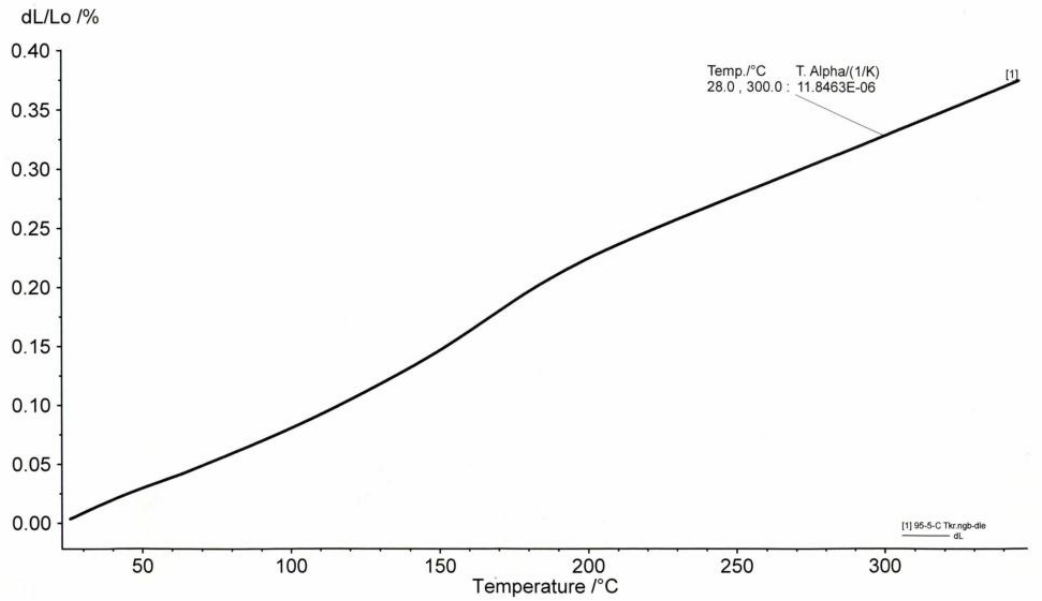


Figure 3.53. Coefficient of thermal expansion (K^{-1}) graph of the foam glass sample with 95 % purity graded SiC (5 wt. %) at 850°C.

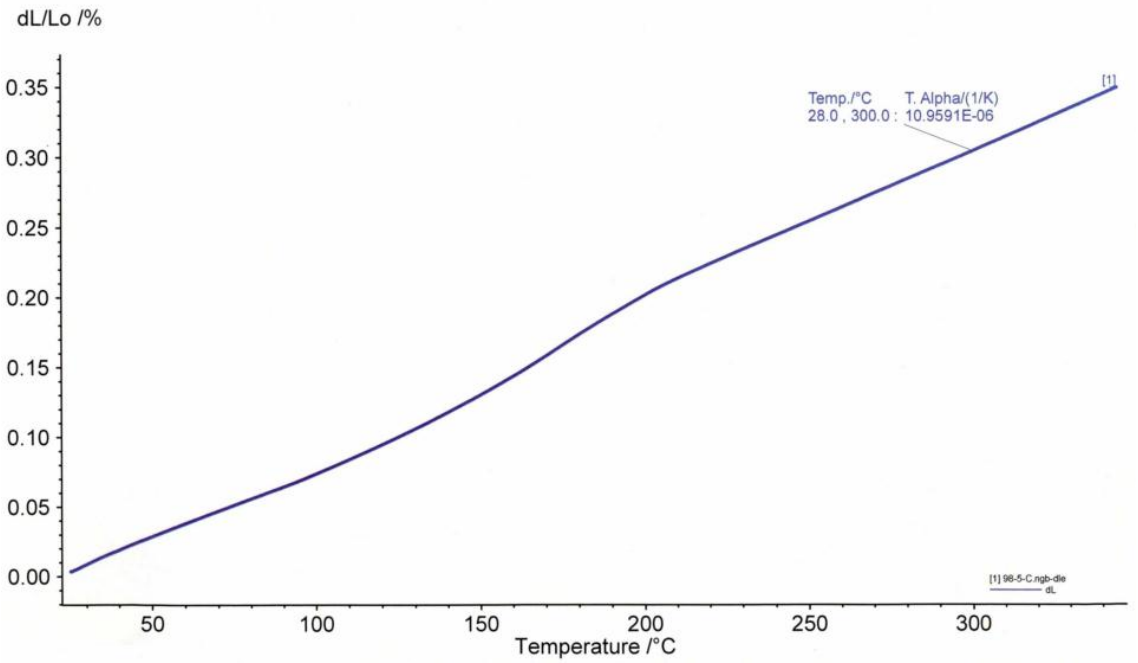


Figure 3.54. Coefficient of thermal expansion (K^{-1}) graph of the foam glass sample with 98 % purity graded SiC (5 wt. %) at 850°C.

The samples produced with 5 wt. % SiC (90, 95 and 98 % purity) and sintered at 850°C were measured to compare the effects of their foaming agent purity grades. All of the samples measured reveal very low thermal expansion coefficient value between 10.95–11.84 x 10^{-6} K^{-1}. Low thermal expansion coefficient may be caused by the raw materials of foam glass. SiC has excellent heat resistant (refractory) properties and it is used for some applications for heat resistance because of having high melting point and very low thermal expansion coefficient like 4.0 x 10^{-6} K^{-1} [80]. Also, commercial soda lime glasses has very low thermal expansion coefficient like 8.9 x 10^{-6} K^{-1} [81]. Thus, the foam glass products can provide low thermal expansion values.

The lowest thermal expansion value was measured with 98 % purity graded SiC foam glass because of the increase in purity grade, hence improved dimensional stability and lower thermal expansion values obtained by higher purity of SiC. Moreover, the results showed that these foam glass materials are proper to use at high temperature applications, because a satisfactory dimensional stability was obtained which is essential for insulation materials without distortion or shrinking.

3.3.11. Water vapor diffusion resistance

Water vapor diffusion resistance defines a material's resistance to the water vapor content passing through unit area in a unit time under specified temperature, humidity and thickness conditions. Resistance to diffusion mechanism occurred in the walls of the buildings is specified with the unit "μ" and every material has a coefficient of water vapor diffusion resistance (μ). This value describes the resistance of the materials to water vapor transfer relative to air. Some materials' μ values were given in Table 3.20.

Table 3.20. Water vapor diffusion resistance (μ) values of some materials [2, 24].

Matter	Coefficient of water vapor diffusion resistance (μ)
Air	1
Fiber glass and stone wool	1
EPS	20-100
XPS	80-250
Foam glass	>40000
Glass	1000000
Metals	1000000

Condensation which is resulted in from the diffusion of water vapor acts negatively on the building materials and heat transfer. Condensation or swelling behavior (rises in winter season in building materials) generates when the surface temperature of the building material in contact with air falls below the temperature of the air. If the water vapor does not condense on the surface of the building material, it enters the building material without sweating. The condensation begins when the partial pressure of the water vapor diffusing into the building material is equal to the water vapor saturation pressure at that temperature at any point inside the layer. Condensation in the material increases the moisture of the material. If it is more than the saturation moisture that the material can absorb, the amount of condensing water is released and moves in the material in various forms. Condensation destroys the structure of construction material, and also increases heat loss with increasing the coefficient of total heat transfer.

Water or moisture, as it is well known, is a very good heat-permeable material and reduces the heat-permeation resistance of the insulation material if it fills capillary air channels or pores in the insulation materials. In addition, insulation material became

deformed as a result of the water or moisture freezing at winter or evaporation at summer seasons.

If a water vapor partial pressure difference occurs between the indoor environment and the outdoor environment in a porous building material, the water vapor passes through the building element towards the side where the partial pressure is high to the side where the partial pressure is low by diffusion. When the vapor that exceeds the boundary layer due to the pressure difference contacts with a surface below the dew point temperature, it causes some condensation on the surface of the building material to sweat, and the remaining water diffuses into the building material. If the water vapor does not condense on the surface of the building material, it enters the building material without sweating. Condensation begins when the partial pressure of the water vapor diffusing into the building material is equal to the water vapor saturation pressure at that temperature at any point in the inner layers. If the temperature in the condensation area is below 0°C, the condensing water vapor transforms into the solid phase. On the other hand, non-condensing water vapor releases from outer surface of the building material and mixes with the outside air [82].

To investigate of one sample's water vapor diffusion resistance behavior, the sample coded as 90-5-C (produced with using 5 wt. % SiC (90 % purity grade) and sintered at 850°C) was analyzed because having most suitable properties like low density and low thermal conductivity. Figure 3.55 represents the measured sample and the results of the test were illustrated in Table 3.21.



Figure 3.55. The foam glass samples (90-5-C) used for water vapor resistance analysis.

Table 3.21. The results of the water vapor diffusion resistance (μ) test.

Parameter	Sample 90-5-C
Ambient temperature (T), (°C)	24.50
Ambient relative humidity (Q), (%)	53.50
Water vapor transmission (kg/(m^2.sec.Pa))	$2.35x10^{-12}$
Water vapor permeability of sample 90-5-C (δ), (kg/(m.sec.Pa))	$3.94x10^{-14}$
Water vapor permeability of air (δ_a), (kg/(m.sec.Pa))	$1.94x10^{-10}$
Water vapor flow density (g/(m^2h))	0.122
Coefficient of the water vapor diffusion resistance (μ)	4920

The coefficient of water vapor diffusion resistance (μ) of the sample 90-5-C was found as quite high with a value of 4920. Matrix structure of the sample was specified as viscous-glassy phase because of being a glass foam material. Thus, the sample highly resisted to water vapor diffusion because of the primary raw material was high amount of glass. However, some transition zones were observed at minimal levels; thus, this is one of the reasons why μ value of the sample isn't as high as required in the related standard. Another reason of being higher of the sample's μ value may result from the mineralogical phases. Commercial foam glass materials are produced with using carbon, carbon black etc. usually. Thus, amorphous phases should dominate the overall foam glass products structure because any carbon base product doesn't remain on the matrix. Since production process in this work is conducted above 700°C, all remaining carbon transforms into CO or CO_2 at this elevated temperature. In the present study, similar production process was applied but the foaming agent was SiC which has very high melting point like 2830 °C. Foaming process starts at nearly 700°C but there should be remain some unreacted SiC after finishing of the process. Also, the XRD phases showed that unreacted SiC remained on the foam glass structures. In addition, some crystalline phases like moissanite, diopside and akermanite generated from the foaming reactions. These reaction products and remaining phases beside amorphous phases of glass may negatively affect the resistance of water vapor diffusion. Nevertheless, the measured μ value is still quite higher rather than other commercial insulation materials like XPS, EPS etc. The foam glass material produced in this work can supply high amount of resistance to water vapor diffusion.

3.4. General Results and Discussion

When characterization results were evaluated in detail, the following results were achieved:

All of the samples volumetrically expanded and transformed into foam glass material. There were three different parameters to control the foaming degree: foaming agent addition ratio (wt. %), SiC purity grade (%) and sintering temperature (°C).

- **Foaming agent addition ratio** dominantly leads the foaming degree of the products. By increasing of foaming agent addition, foaming process accelerated because higher amount of reducing reaction and gaseous species occurred. Also, growing of voids within the cells resulted in density and thermal conductivity reduction because of porosity enhancement. In general, 5 wt. % was determined as optimum foaming agent addition ratio; because, some diverge from the desired properties was obtained below and above this ratio. Low amount of addition could not maintain required foaming; whereas, above the 5 wt. % additions, density increasing and pore size reduction occurred especially at samples produced with 90 and 95 % purity grade SiC. On the other hand, foaming process was enhanced with above 5 wt. % addition of 98 % purity graded SiC. This may because from the high purity SiC hardly reacted and it supplied the required foaming with higher addition ratios.
- **Foaming agent purity grade** significantly influenced the foaming process. Lower purity grade SiC could provide improved foaming reactions because of its lower melting point and some impurity content which lead to easy reaction at lower temperatures. On the other hand, with the higher purity graded SiC foaming agent, more homogeneous cell structures were achieved due to capability of generating more homogeneous gaseous product and there wasn't a sufficient time for enlargements of the pores. Also, a prominently reduction was revealed in pore sizes with the higher purity graded SiC. The lower the purity graded SiC caused the larger cell sizes; similarly, the higher the purity graded SiC, the smaller the cell size was obtained. This is the reason of achieving higher strength, higher density and higher thermal conductivity values with the increasing of SiC purity grade.

- **Sintering temperature** directly affects to foam of glass. Increasing of temperature at 850°C caused increasing of volume expansion which means that higher porosity and lower density. Moreover, lightweight samples gave very low thermal conductivity values. However, slight decrease in density resulted in compression strength reduction, or any increasing in density leads higher strength. Above 850°C, some collapsing and tearing on the cell wall began. Because of this distortion of the samples sintered at 875°C, shrinkage became evident and porosity significantly declined. This resulted in rising of density, strength, and water absorption but lower thermal conductivity because of starting degradation and interconnection of pores.

- It can be concluded that the optimum sample 90-5-C which was produced with using 90 % purity graded SiC with a ratio of 5 wt. % and sintered at 850°C. The highest volume expansion ratio (802.48 %) and the lowest density (1.6 g/cm^3) were achieved from this sample. 90-5-C revealed very high porosity (76.77 %) and very low thermal conductivity value (0.046 W/mK) as a result of this high porosity and low density. Compressive strength value (1.50 MPa) and water vapor diffusion resistance (4920 μ) were found sufficient. Finally, the sample 90-5-C can be preferred as an insulation material.

- Also, some other samples provided suitable results close to the 90-5-C. According to the raw material and sintering temperature preference, various alternative foam glass formulas were developed and represented to the producer choice. Table 3.22 illustrates the general results of the characterization of selected foam glass samples according to their foaming agent purity grade, addition ratio and sintering temperature. Also, the values specified in the standard TS EN 13167:2013-04 and commercial foam glass products were given in the table for comparison.

Table 3.22. Comparison of physical properties of selected foam glass samples with standard value [24, 31] and commercial product [32].

Foam glass product information			Volume expansion ratio (%)	Compressive strength (MPa)	Apparent density (g/cm^3)	Long term water absorption ratio (%)	Thermal conductivity (W/mK)
SiC foaming agent purity grade (%)	SiC foaming agent addition ratio (wt. %)	Sintering temperature (°C)					
90	1	850	475.73	4.23	0.251	65.72	0.076
90	5	850	802.48	1.50	0.164	56.71	0.046
90	9	850	759.29	1.23	0.174	35.31	0.052
90	5	800	692.75	2.18	0.182	18.13	0.048
90	5	825	759.57	1.70	0.169	24.05	0.045
90	5	875	681.40	1.72	0.188	57.09	0.047
95	5	850	375.40	2.61	0.304	23.31	0.057
98	5	850	320.19	8.38	0.358	43.71	0.065
TS EN 13167:2013-04			-	≥0.4 - 3	0.090 – 0.180	<0.5	0.045 – 0.060
Pittsburgh Corning Foam glass			-	0.62	0.100 – 0.160	<0.2	0.039 – 0.050

Finally, characterization results revealed some correlations between the properties of foam glass samples and following conclusions were drawn:

- Volume expansion ratio dominated the most important features of foam glasses. The higher volume expansion ratio leads to the lower apparent density.
- However, compressive strength reduction occurred with decreasing of density values. Higher density with lower volume expansion caused stronger foam glass in terms of compressive strength.
- Figure 3.56, shows the variation of the volume expansion effect on the apparent density and compressive strength of foam glass samples.

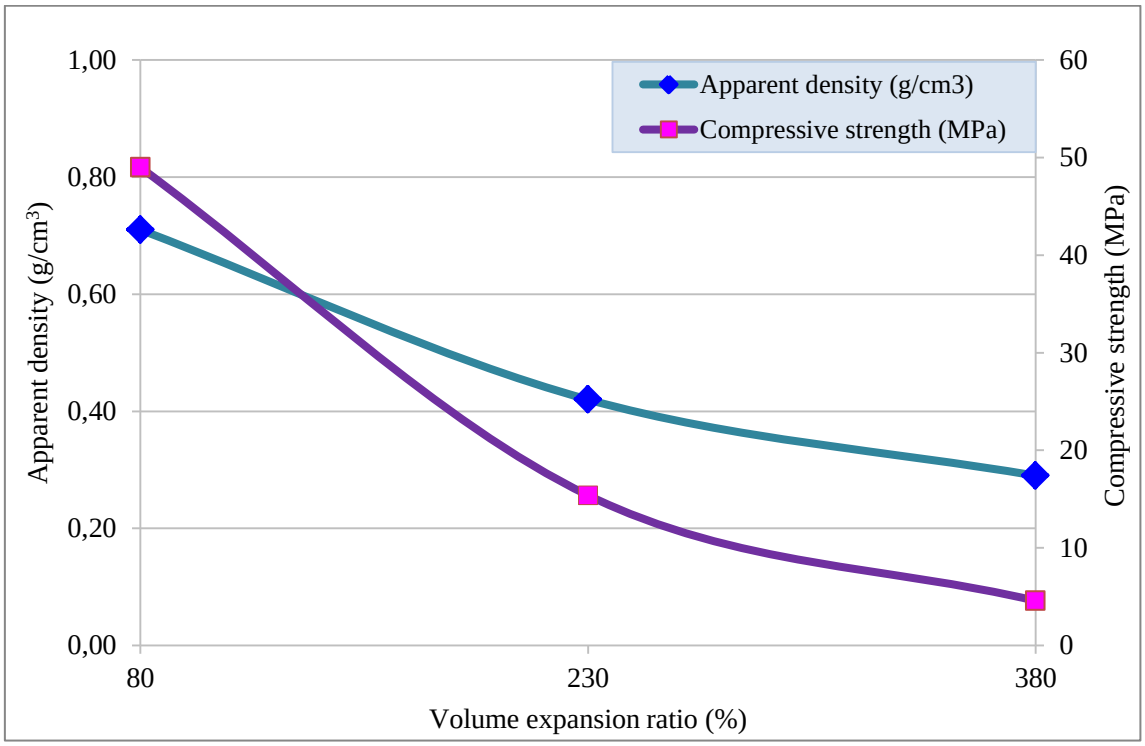


Figure 3.56. Volume expansion - apparent density- compressive strength correlation graph of foam glasses.

- Volume expansion ratio also affected total porosity values of foam glass samples.
- During expansion of glass, large amount of voids were occurred within the cell structures and this caused declining of the apparent density.
- Figure 3.57 represented the correlation between total porosity and density values corresponding to volume expansion.

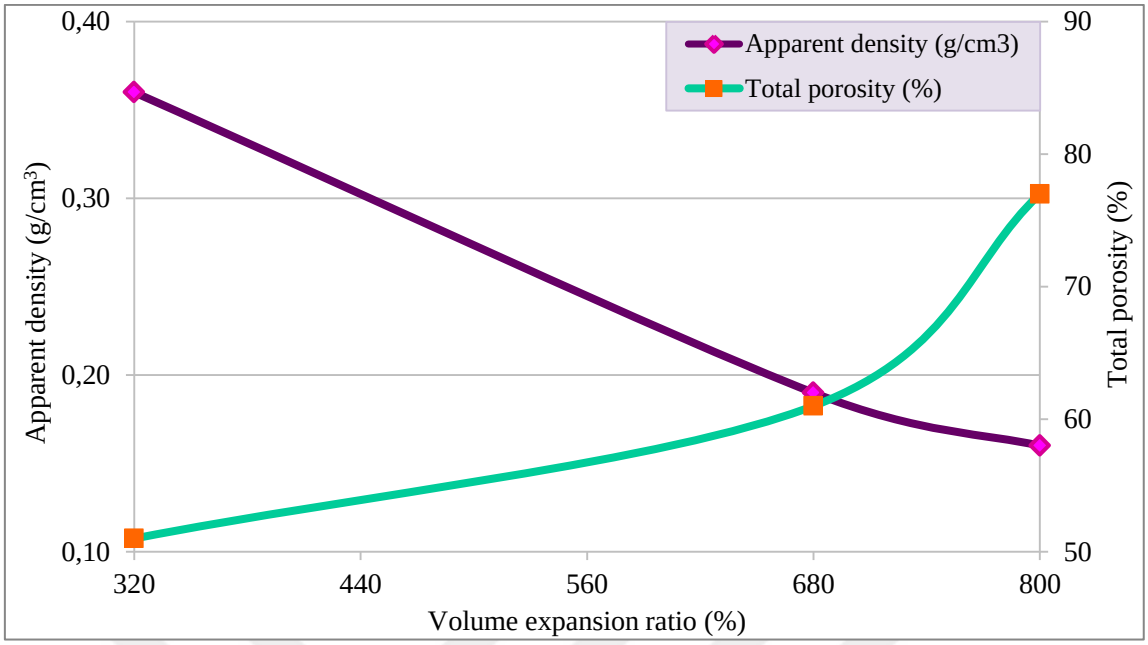


Figure 3.57. Volume expansion - apparent density- total porosity correlation graph of foam glasses.

- Moreover, high porosity provides higher thermal insulation property. Porosity increase prevented the heat transfer and provided lower thermal conductivity value beside of reducing density.
- Porosity effect on the density and thermal conductivity values of foam glass can be seen in Figure 3.58.

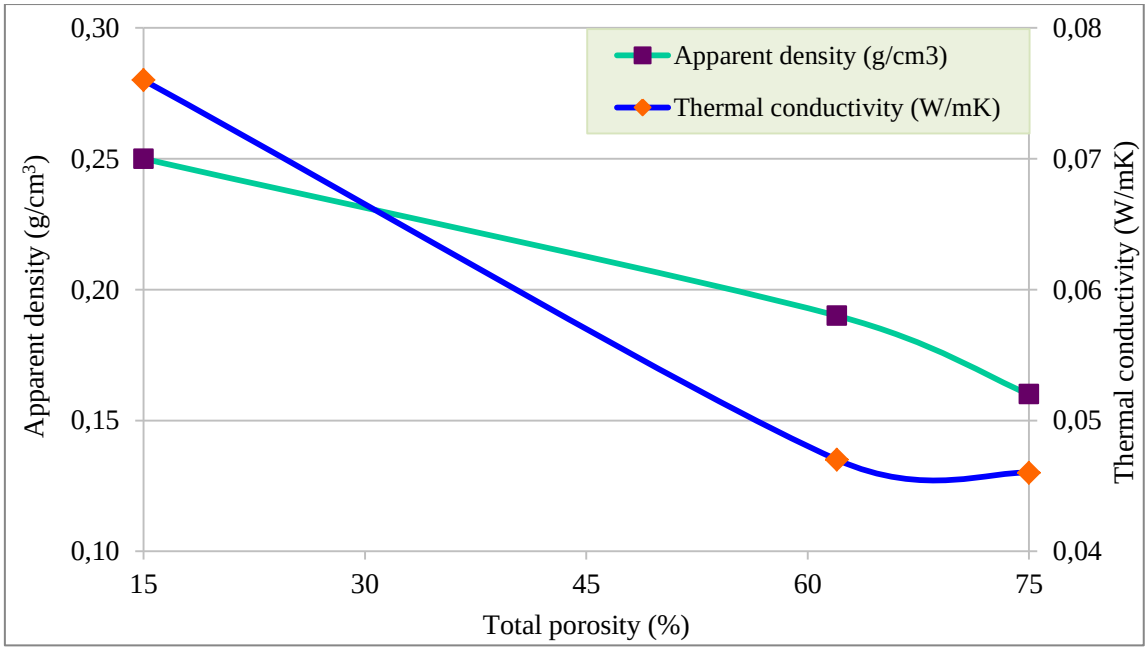


Figure 3.58. Total porosity- apparent density - thermal conductivity correlation graph of foam glasses.

CHAPTER 4 - CONCLUSION

In the present study, effects of some parameters on foam glass properties were investigated. Within this scope, three main parameters were researched and analyzed: foaming agent addition ratio, foaming agent purity grade and sintering temperature.

Three different purity graded SiC (90, 95 and 98 % purity graded) were used as foaming agent with a ratio of 1, 3, 5, 7 and 9 wt. % and waste sheet glass powder as matrix material. The waste glass-SiC mixture was then sintered at 800, 825, 850 and 875°C with same heating rate and holding time. Sintered samples were characterized by measuring volume expansion ratio, XRD mineralogical analysis, apparent density, compressive strength, short and long term water absorption, SEM examination, thermal conductivity, total porosity, thermal expansion coefficient and water vapor diffusion resistance.

Foam glass samples met the required properties which were declared in the standard TS EN 13167:2013-04. Also, the following results were achieved from the characterization study:

- Increase in foaming agent addition accelerated the expansion of glass-SiC mixture and caused forming of a cellular glass. In general, high amount of SiC addition lead to more porosity, lower density and lower thermal conductivity. In general, 5 wt. % SiC supplied the most proper results among all samples.
- Surprisingly, SiC foaming agent purity grade increasing had a negative effect on foaming process. Lower purity graded SiC agents could create faster and easier foaming reaction at same temperatures because of having lower melting point and containing impurities. Both of the required density and thermal conductivity values could be achieved from only 90 % purity graded SiC foaming glass.
- Temperature increase was a driving force for foaming process. However, some undesired distortion and tearing started on the cell walls with sintering above 850°C. Also, foaming process couldn't be sufficiently obtained below 850°C in many samples. Thus, 850°C was decided as the optimum sintering temperature.

In conclusion, various foam glass products were achieved having low density, low thermal conductivity and high porosity beside their sufficient compressive strength. The

formulas of the developed foam glass can provide an insight for the production as an insulation material. Distinctively from the literature, different purity graded SiC foaming agent was used and relatively lower sintering temperatures between 800-875°C were applied to produce foam glass samples. In comparison to the other studies reported in the literature, it was challenged in this study that 90 % purity graded SiC foaming agent and 850°C sintering temperature supplied the required low density and low thermal conductivity values which were achieved with higher purity SiC and higher sintering temperatures at the literature. The study also showed that a suitable usage or recycling area for waste glass because of the capability of its transforming from a waste into a valuable insulation material. Since a promising insulting material was produced as an outcome this study, this work can be an example to evaluate high amount of glass waste as primary raw material, and can provide an effective solution for waste glass utilization problem. Moreover, an alternative insulation material was presented for using in the building insulation which is essential for energy efficiency in recent years. This study suggested that foam glass materials can be produced with desired properties and can be used at various insulation systems rather than importing polymer based unhealthy insulation materials.

FUTURE WORK

The present study demonstrated that the effects of foaming agent purity grade, composition ratio and sintering temperature on foam glass characteristic properties. From the literature knowledge and experimental studies, it was observed that features of foam glass are quite sensitive to any change on raw material or sintering schedule. Thus, it may be investigated the influence of the raw materials grain size, mineral additives, altering glass sources, altering foaming agent sources, mixing time of raw materials, heating rate, holding time at peak temperature and cooling rate. All of these parameters can modify the foam glass structure and required properties. In other words, the influence of these variables on production of foam glass may investigate to be suggested as future works.

REFERENCES

[1] Özutku, O., Karakuş, C. (2011) Binalarda Isı Yalıtımı Yoluyla Enerji Tasarrufu ve Örnek Bir Uygulama: MKÜ Mühendislik Fakültesi Binasının Enerji Performans Değerleri ve Maliyetleri. X. Ulusal Tesisat Mühendisliği Kongresi, 13-16 Nisan, İzmir, Türkiye.

[2] Yaman, Ö., Şengül, Ö., Selçuk, H., Çalıkuş, O., Kara, İ., Erdem, Ş., Özgür, D. (2015) Binalarda Isı Yalıtımı ve Isı Yalıtım Malzemeleri. Türkiye Mühendislik Haberleri (TMH), 487(4), 62-75.

[3] http://www.izoder.org.tr/dosyalar/dosemelerde_isiyalitimi.pdf (01.05.2017).

[4] https://www.gnyapi.com.tr/u-degeri-hesaplama (01.05.2017).

[5] Scarinci, G., Brusatin, G., Bernardo, E. (2005) Glass Foams, Cellular Ceramics: Structure, Manufacturing, Properties and Applications, Editors.; Scheffler, M., Colombo, P., 158-176, WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim, Germany.

[6] Kulaksızoğlu, Z. (2006) Isı Yalıtım Sektör Araştırması, İstatistik Şubesi.

[7] TS 825 (2008) Thermal Insulation Requirements for Buildings. Turkish Standards Institutes, Ankara, Turkey.

[8] https://www.thenbs.com/knowledge/what-is-a-u-value-heat-loss-thermal-mass-and-online-calculators-explained (01.05.2017).

[9] http://www.pakboard.com.tr/index.asp?action=pakboard_eps_xps_karsilastirma (25.04.2017)

[10] http://www.xpsturkiye.org/sayfa.asp?ID=129 (25.04.2017)

[11] http://www.kalekim.com/Urunler.asp?ProdGroupID=22 (25.04.2017)

[12] http://www.izocam.com.tr/f1-camyunu.html# (25.04.2017)

[13] http://www.izocam.com.tr/f2-tasyunu.html (25.04.2017)

[14] http://www.purtek.com.tr/poliuretan-sert-kopuk-nedir.html (25.04.2017)

[15]http://www.assanpanel.com.tr/~/media/files/pdf/quality/malzeme-pektleri/poliüretan.ashx (25.04.2017)

[16] https://www.guneyyapiizolasyon.com.tr/sayfalar.3988.fenol-kopugu.html (25.04.2017)

[17] https://www.gnyapi.com.tr/fenol-kopugu-nedir (25.04.2017)

[18] http://www.kuranelyapi.com/sayfa/isi-yalitim-malzemeleri,64.php (04.05.2017)

[19] http://perpan.com.tr/urunlerimiz4ef9.html?id=12 (04.05.2017)
[20] https://www.gnyapi.com.tr/genlestirilmis-prlit (04.05.2017)
[21] https://www.gnyapi.com.tr/genlestirilmis-mantar (04.05.2017)
[22] http://www.bina.com.tr/32/33/Tr/isi-yalitimi-nedir.html (04.05.2017)
[23] https://www.gnyapi.com.tr/cam-kopugu-nedir (04.05.2017)
[24] TS EN 13167:2013-04 (2013) Thermal Insulation Products for Buildings - Factory Made Cellular Glass (CG) Products–Specification. Turkish Standards Institutes, Ankara, Turkey.
[25] http://www.isopa.org/news/the-benefits-and-history-of-polyurethane-flexible-foam/ (02.06.2017)
[26] http://www.unitechenterprise.in/insulation-boards.html (02.06.2017)
[27] http://www.archiexpo.com/prod/foamglas/product-71744-1365735.html (02.06.2017)
[28] Hurley, J. (2003) A UK Market Survey for Foam Glass. Glass Development and Research Final Report, WRAP Creating Markets for Recycled Resources, GLA-0015, Published by: The Waste and Resources Action Programme, Oxon, England.
[29] http://www.teknosel.com/urun.php?u=22&c=3 (04.05.2017)
[30]https://www.google.com.tr/search?q=foam+glass&source=lnms&tbm=isch&sa=X&sqi=2&ved=0ahUKEwiI6677xprUAhXG8RQKHTOUAw0Q_AUIBigB&biw=1920&bih=964#imgrc=SUkZKGxGCb40mM:&spf=1496248219492 (04.05.2017)
[31] BS EN 13167:2012+A1:2015 (2015) Thermal Insulation Products for Buildings — Factory Made Cellular Glass (CG) Products— Specification. British Standards Institutes, UK.
[32] http://www.foamglas.com/ (02.06.2017)
[33] ASTM C518-15 (2015) Standard Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus. ASTM International, West Conshohocken, PA. https://doi.org/10.1520/C0518-15.
[34] ASTM C177-13 (2013) Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus. ASTM International, West Conshohocken, PA. https://doi.org/10.1520/C0177.
[35] ISO 13787 (2003) Thermal Insulation Products for Building Equipment and

Industrial Installations - Determination of Declared Thermal Conductivity. International Organization for Standardization. https://www.iso.org/standard/37056.html.

[36] ASTM C165-07 (2012) Standard Test Method for Measuring Compressive Properties of Thermal Insulations. ASTM International, West Conshohocken, PA. https://doi.org/10.1520/C0165-07R12.

[37] ASTM C240-16 (2016) Standard Test Methods of Testing Cellular Glass Insulation Block. ASTM International, West Conshohocken, PA. https://doi.org/10.1520/C0240-16.

[38] ASTM C552-16a (2016) Standard Specification for Cellular Glass Thermal Insulation. ASTM International, West Conshohocken, PA. https://doi.org/10.1520/C0552-16A.

[39] TS EN 826:2013-06 (2013) Thermal Insulating Products for Building Applications-Determination of Compression Behaviour. Turkish Standards Institute, Ankara, Turkey.
https://intweb.tse.org.tr/standard/standard/Standard.aspx?081118051115108051104119110104055047105102120088111043113104073086055088102113081053084107049090.

[40] ASTM C203-05a (2012), Standard Test Methods for Breaking Load and Flexural Properties of Block-Type Thermal Insulation. ASTM International, West Conshohocken, PA. https://doi.org/10.1520/C0203-05AR12.

[41] TS EN 12089 (2013) Thermal Insulating Products for Building Applications-Determination of Bending Behavior. Turkish Standards Institute, Ankara, Turkey.

[42] ASTM C623-92 (2015) Standard Test Method for Young's Modulus, Shear Modulus, and Poisson's Ratio for Glass and Glass-Ceramics by Resonance. ASTM International, West Conshohocken, PA. https://doi.org/10.1520/C0623-92R15.

[43] TS EN 1602:2013-06 (2013) Thermal Insulating Products for Building Applications-Determination of Apparent Density. Turkish Standards Institute, Ankara, Turkey.

[44] TS EN 1609:2013-06 (2013) Thermal Insulating Products for Building Applications-Determination of Short Term Water Absorption by Partial Immersion. Turkish Standards Institute, Ankara, Turkey.

[45] TS EN 12087:2013-06 (2013) Thermal Insulating Products for Building

Applications-Determination of Long Term Water Absorption by Immersion. Turkish Standards Institute, Ankara, Turkey.

[46] ASTM E96 / E96M-16 (2016) Standard Test Methods for Water Vapor Transmission of Materials. ASTM International, West Conshohocken, PA. https://doi.org/10.1520/E0096_E0096M-16.

[47] TS EN 12086:2013-06 (2013) Thermal Insulating Products for Building Applications - Determination of Water Vapour Transmission Properties. Turkish Standards Institute, Ankara, Turkey.

[48] TS EN ISO 12572:2001-11 (2001) Hygrothermal Performance of Building Materials and Products - Determination of Water Vapour Transmission Properties. Turkish Standards Institute, Ankara, Turkey.

[49] ASTM E84-16 (2016) Standard Test Method for Surface Burning Characteristics of Building Materials. ASTM International, West Conshohocken, PA. https://doi.org/10.1520/E0084-16.

[50] TS EN ISO 1182:2010-07 (2010) Reaction to Fire Tests for Products - Non-Combustibility Test. Turkish Standards Institute, Ankara, Turkey.

[51] ASTM C411-11 (2011) Standard Test Method for Hot-Surface Performance of High-Temperature Thermal Insulation. ASTM International, West Conshohocken, PA. https://doi.org/10.1520/C0411-11.

[52] TS EN 1604:2013-06 (2013) Thermal Insulating Products for Building Application Determination of Dimensional Stability Under Specified Temperature and Humidity Conditions, Turkish Standards Institute, Ankara, Turkey.

[53] ASTM E228-17 (2017) Standard Test Method for Linear Thermal Expansion of Solid Materials with a Push-Rod Dilatometer. ASTM International, West Conshohocken, PA. https://doi.org/10.1520/E0228-17.

[54] BS EN 13471:2001 (2001) Thermal Insulating Products for Building Equipment and Industrial Installations. Determination of the Coefficient of Thermal Expansion. British Standards Institution.
https://shop.bsigroup.com/ProductDetail/?pid=000000000030049964.

[55] FOAM GLAS Insulation Systems for Industrial Applications (2014) Pittsburgh Corning UK Ltd., Technical Data Sheet.

[56] FOAM GLASS Insulation Systems for Industrial Applications with Operating

Temperatures of -450°F to 900°F (-268°C to 482°C). Pittsburgh Corning UK Ltd., Technical Data Sheet.

[57] FOAM GLASS Interior Insulation Systems. Pittsburgh Corning UK Ltd., Technical Data Sheet.

[58] http://www.archello.com/en/product/foamglas%c2%ae-cellular-glass-insulation (05.06.2017)

[59] Tulyaganov, D.U., Fernandes, H.R., Agathopoulos, S., Ferreira, J.M.F. (2006) Preparation and Characterization of High Compressive Strength Foams from Sheet Glass. J Porous Mater, Vol. 13, 133–139.

[60] Bayer, G. (1980) Foaming of Borosilicate Glasses by Chemical Reactions in the Temperature Range 950 - 1150°C. Journal of Non-Crystalline Solids, 38&39, 855-860.

[61] Ercenk, E. (2017) The Effect of Clay on Foaming and Mechanical Properties of Glass Foam Insulating Material. Journal of Thermal Analysis and Calorimetry, Vol.127, Issue 1, 317-146. (DOI 10.1007/s10973-016-5582-8).

[62] Méar, F., Yot, P., Cambon, M., Ribes, M. (2005) The Changes in Lead Silicate Glasses Induced by the Addition of Reducing Agent (TiN or SiC). Journal of Non-crystalline Solids, 351, 3314-3319.

[63] Mugoni, C., Montorsi, M., Siligardi, C., Andreola, F., Lancellotti, I., Bernardo, E., Barbieri, L. (2015) Design of Glass Foams with Low Environmental Impact. Ceramics International, 41, 3400-3408.

[64] Bernardo, E., Cedro, R., Florean, M., Hreglich, S. (2007) Reutilization and Stabilization of Wastes by the Production of Glass Foams. Ceramics International, 33, 963-968.

[65] Méar, F., Yot, P., Ribes, M. (2006) Effects of Temperature, Reaction Time and Reducing Agent Content on the Synthesis of Macroporous Foam Glasses from Waste Funnel Glasses. Materials Letters, 60, 929-934.

[66] König, J., Petersen, R.R., Yue, Y. (2014) Influence of the Glass–Calcium Carbonate Mixture's Characteristics on the Foaming Process and the Properties of the Foam Glass. Journal of European Ceramic Society, 34, 1591-1598.

[67] Spiridonov, Y.A., Orlova, L.A. (2003) Problems of Foam Glass Production. Glass and Ceramics, Vol.60, No.10, 313-314.

[68] Brusatin, G., Bernardo, E., Scarinci, G. (2004) Production of Foam Glass from

Glass Waste, Sustainable Waste Management and Recycling: Glass Waste, Editors; Limbachiya, M.C, Roberts, J.J., 67-82, London, UK.

[69] Guo, H.W., Gong, Y.X., GaO, S.Y. (2010) Preparation of High Strength Foam Glass-Ceramics From Waste Cathode Ray Tube. Materials Letter, 64, 997-999.

[70] Qu, Y.N., Huo, W.L., Xi, X.Q., Gan, K., Ma, N., Hou, B.Z., Su, Z.G., Yang, J.L. (2016) High Porosity Glass Foams from Waste Glass and Compound Blowing Agent. Journal of Porous Materials, Vol.23, Issue 6, 1451-1458. DOI: 10.1007/s10934-016-0205-0.

[71] Hasheminia, S., Nemati, A., Yekta, B.E., Alizadeh, P. (2012) Preparation and Characterisation of Diopside-Based Glass-Ceramic Foams. Ceramics International, 38, 2005-2010.

[72] Samoilenko, V.V., Uglova, T.K. (2016) Kinetics of Foam Glass Formation with Different Heating Rates. Glass and Ceramics, Vol.73, Nos. 7-8, 237-239. DOI 10.1007/s10717-016-9864-1.

[73] Méar, F., Yot, P., Viennois, R., Ribes, M. (2007) Mechanical Behaviour and Thermal and Electrical Properties of Foam Glass. Ceramics International, 33, 543–550.

[74] Méar, F., Yot, P., Cambon, M., Caplain, R., Ribes, M. (2006) Characterisation of Porous Glasses Prepared from Cathode Ray Tube (CRT). Powder Technology, 162, 59-63.

[75] Francis, A.A., Rahman, M.K.A. (2013) Experimental Design for Optimisation of Density and Water Absorption Capacity of Glass–Ceramic Foams Prepared from Silica Rich Wastes. Powder Metallurgy, Vol. 56, No. 4, 295-303.

[76] http://merlab.metu.edu.tr/parcacik-boyut-analiz-cihazi (13.06.2017).

[77] Sözbir, N. (2014) Isı İletim Katsayısının Belirlenmesi Deneyi. Makina Mühendisliği Laboratuvarı II Dersi, Sakarya.

[78] https://me-mechanicalengineering.com/fouriers-law/ (13.06.2017)

[79] http://merlab.metu.edu.tr/civa-porozimetresi (13.06.2017)

[80] http://accuratus.com/silicar.html (21.06.2017)

[81] http://www1.udel.edu/chem/GlassShop/PhysicalProperties.htm (21.06.2017)

[82] Heperkan, H.A., Bircan, M.M., Sevindir, M.K. (2001) Yapı Malzemelerinde Buhar Difüzyonu ve Yoğuşma. V. Ulusal Tesisat Mühendisliği Kongresi ve Sergisi, 3-6 October, İzmir.

APPENDIX 1 – Table 1. Literature survey with key points.

Author Name	Raw Material			Particle Size	Mixture Ratio	Batch Mixing Time	Press Pressure	Sintering Shedule	Results
	Glass	Foaming Agent	Mineral or Additive						
Tulyaganov et al. (2006)	Sodium-calcium-silicate sheet glass (C) and alkali earth aluminosilicate glass (AD)	SiC		C= 9 µm AD= 13 µm SiC= 17 µm	• C= 89-94 wt. % • AD= 5-10 wt. % • SiC= 1 wt. %	30 min	-	Heating from 25 to 900°C (5 K/min), holding for 30 minutes at peak temperature, heating from 900 to 950°C (5 K/min) and holding for 30 minutes	• Bulk density= 0.252-0.309 g/cm^3 • Porosity= 87.90-90.05 % • Compressive strength= 2.132-2.601 MPa
Bayer (1980)	Borosilicate glass	SiC and Si_3N_4	CuO Kaolin	Glass= <0.1 mm Foaming agents= <0.01 mm Minerals= <0.05 mm	• SiC or Si_3N_4=5 wt. % • Kaolin= 10-30 wt. %			Heating from 25 to 950-1150ºC at 2 hours, holding at peak temperature for 5 minutes and very slow cooling with 0.5-2ºC/min.	• Volume expansion= 10 times • Bulk density= 0.1 g/cm^3
Méar et al. (2007)	Funnel glass, panel glass and 2:1 mixture of funnel and panel glass	SiC or TiN	Arabic gum as binder	TiN= <10 µm SiC= <63 µm Glass= <63 µm	• TiN= 4 wt. % • SiC= 5 wt. %		5 tonnes	750ºC for 120 minutes	• Thermal conductivity= 0.08–0.43 W m^{-1} K^{-1} • Compressive strength= 4-267 MPa • Flexural strength= 4-68 MPa • Porosity= 84.5-86.1 %
Bernardo et al. (2007)	Recycled soda-lime glass waste	- SiC-based wastes coming from the polishing of glass articles (Contains about 50 wt. % SiC)	MnO_2	Glass= <37 µm SiC= <50 µm MnO_2= 10 µm	• SiC based polishing waste= 2.5 - 17.5 wt. % • MnO/SiC-based waste= 1/5 or 1/3	30 min		Heating to 950°C, (10°C/min), holding for 1 h, drastically cooled (more than 10°C/min rate) to 600°C, then slowly cooling (≈1 8°C/min) to 500°C, and again fast cooling (≈5°C/min) to room temperature.	• Relative density= ϕ=0.08 • Porosity= 92 %

APPENDIX 1 – Table 1. Literature survey with key points (Continued).

Author Name and Year	Raw Material			Particle Size	Mixture Ratio	Batch Mixing Time	Press Pressure	Sintering Shedule	Results
	Glass	Foaming Agent	Mineral or Additive						
Méar et al. (2005)	Lead silicate glasses	TiN or SiC			TiN or SiC= 1, 3, 5, 7 or 9 wt.%			Temperatures between 700-900°C, holding about 90 minutes	High expansion was achieved.
Hasheminia et al. (2012)	Diopside glass-ceramic composition	SiC	PbO	Glass= 4.5 µm SiC= 12 µm	SiC= 2 wt. % PbO= 0, 5, 10, 15 wt. %	15 min	40 MPa	Drying of pellets at 100°C, heating to 850-1000°C (10°C/min) and holding for 1 h.	Bulk density= 0.5 g/cm^3
Méar et al. (2006)	CRT: funnel and/or screen glass	SiC TiN	MgO		SiC= 3.34, 5 and 6.68 wt. % TiN= 2.66, 4.00 and 5.32 wt. % MgO= 2, 3 and 4 wt. %			Sintering at 800°C for 60 min	• Bulk density= 0.51-0.99 g/cm^3 • Skeletal density= 2.53-2.71 g/cm^3 • Open porosity= 63.6 to 81.0 % • Closed porosity= 0.9 to 2.7 % • Total porosity= 65.1 to 82.5 %
Francis et al. (2013)	Granulated blast furnace slag and glass cullet	SiC			Glass cullet= 70 wt. % SiC= 1, 5 and 10 wt. %			• Sintering at 900, 950 or 1000°C • Holding time= 30, 60 or 120 min	• Compressive strength= 1.18 – 1.8 MPa • Density= 0.61 g/cm^3

CURRICULUM VITAE



Name Surname : Aylin ŞAHİN
Address : Kadıköy / İstanbul
Tel (Office) : (262) 677 30 97
GSM : (537) 510 07 88
E-mail : eaylinsahin@gmail.com

EDUCATION

MSc : Metallurgical and Materials Engineering (2014 – 2017)
University : Marmara University, Institute for Graduate Studies in Pure and Applied Sciences
City, Country : Istanbul, Turkey

BSc : Metallurgical and Materials Engineering (2009 – 2014)
University : Marmara University, Engineering Faculty
City, Country : Istanbul, Turkey

WORK EXPERIENCE

Institution : TUBITAK Marmara Research Centre, Materials Institute (2015 – …)
City, Country : Kocaeli, Turkey
Position : Researcher

PUBLICATIONS

a) Journal Publications:

1. Ozalp, F., Yilmaz, H.D., Kara, M., Kaya, Ö., Şahin, A. (2016) Effects of Recycled Aggregates from Construction and Demolition Wastes on Mechanical and Permeability Properties of Paving Stone, Kerb and Concrete Pipes. Construction & Building Materials, Vol. 110, pp. 17-23.
2. Kilic, Y., Kara, M., Demir, B.M., Sahin, A., Unal, C. (2017) The Use of Construction and Demolition Wastes in the Brick Production. International Journal of Recent Trends in Engineering & Research, Vol.3, Issue 5, 569-578.
3. Sahin, A., Artir, R. (2017) Influence of the Different Parameters on Foam Glass Production: Foaming Agent Purity Grade, Compositon Ratio and Sintering Temperature. Ceramics International ***(in editor review)***

b) Conference Publications:

1. Kara, M., Kilic, Y., Sahin, A. (2015) İnşaat ve Yıkıntı Atıklarının Tuğla Üretiminde Kullanılabilirliğinin Araştırılması. 7. Ulusal Katı Atık Yönetimi Kongresi (UKAY 2015), Organized by: Gaziantep Üniversitesi ve KAKAD, 14-16 October, Gaziantep.
2. Kara, M., Kilic, Y., Demir, B.M., Ustaoglu, U., Sahin, A., Unal, C. (2016) Utilization of Construction and Demolition Wastes in Different Industrial Products. EURASIA Waste Management Symposium, 02-04 May, İstanbul.
3. Kara, M., Kilic, Y., Sahin, A., Ziypak, M., Olgun, Z. (2016) Şlam Atıklarının Tuğla Üretiminde Hammadde Olarak Değerlendirilmesi. 20. Kömür Kongresi, 4-6 May, Zonguldak.
4. Kara, M., Kilic, Y., Sahin, A., Demir, B.M. (2016) Tuğla ve Kiremit Atıklarının Tuğla Üretiminde Kullanılma Potansiyeli. 8. Ulusal Katı Atık Yönetimi Kongresi (UKAY 2016), 11-14 May, Karabük.
5. Kilic, Y., Kara, M., Sahin, A., Demir, B.M., Ozalp, F., Yilmaz. H. (2016) Kentsel Dönüşüm ve Uygulaması. 1. Uluslararası Şehir, Çevre ve Sağlık Kongresi, 11-15 May, KKTC.
6. Ziypak, M., Olgun, Z., Sahin, A., Kilic, Y., Kara, M. (2017) An Alternative Raw Material in Brick Production: Coal Slurry Waste. 25th International Mining Congress (İMCET 2017), pp.514-523, 11-14 April, Antalya.
7. Ziypak, M, Olgun, Z., Turan, M., Erdogan, J., Kilic, Y., Sahin, A., Kara, M. (2017) Utilization of Coal Slurry Waste as an Alternative Raw Material in Portland Cement Clinker Production. 5th International Conference on Sustainable Solid Waste Management, 21–24 June, Athens.