STRUCTURAL AND SURFACE PROPERTIES OF ZINC-TELLURITE GLASS DOPED WITH SILICON DIOXIDE

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To my beloved mother, step father, siblings, grandma, aunts, uncles, cousins, late father who was very proud of me and other family members

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ABSTRACT

This thesis studied the influence of silicon dioxide (SiO_2) on the physical, thermal, structural and surface properties of zinc-tellurite glass. A series of transparent ternary tellurite glass systems with composition (80-*x*)TeO_2–20ZnO–*x*SiO_2, where *x* = 0 till 0.20 mol% were synthesized via conventional melt-quenching method. The effects of varying SiO_2 contents on the glass density, structural characteristics, thermal properties, water contact angle (WCA), surface roughness and surface morphology were investigated. The amorphous nature of the glass was confirmed using X-ray diffraction measurement. The increase in the SiO_2 contents reduced the density from 5.55 to 5.53 $g.cm^{-3}$ and enhanced the molar volume from 25.96 to 26.06 $cm^3.mol^{-1}$. Differential thermal analysis revealed the increase of glass transition temperature (T_g) with the increase of SiO_2 contents. The thermal stability factor was found to vary between 79.9 to 81.5°C depending on the glass composition. Glass morphologies were characterized using atomic force microscopy and WCA measurements. Glass surface roughness as much as 10 nm was attained which is higher than normal glass. The optimal value for WCA was discerned to be 101° and the minimum surface energy was found to be 0.06 $N.m^{-1}$ for glass sample with 0.1 mol% of SiO_2 indicating a glass with low wettability compared to other zinc-tellurite glass. The structural characteristics of glass samples were investigated using Fourier transform infrared (FTIR) spectroscopy in the range of 600 – 4000 cm^{-1}. The FTIR results showed the tellurium atom coordination changes from TeO_4 to TeO_3 with the increase of SiO_2 contents. For TeO_4 coordination, the infrared band peaks occurred between 642.8 and 661.7 cm^{-1} whereas for TeO_3, the peaks were observed between 784.7 and 773.1 cm^{-1}. Field emission scanning electron microscopy and energy dispersive X-ray spectroscopy were performed on the glass sample. The surface morphology of glass system appeared rougher with the addition of SiO_2 content up to 0.1 mol% which is better compared to normal glass. Overall, the physical, thermal and structural properties of the proposed zinc-tellurite glass system were marginally changed while the surface properties were significantly improved with the inclusion of SiO_2.

ABSTRAK

Tesis ini mengkaji tentang pengaruh silikon dioksida (SiO_2) terhadap sifat fizikal, terma, struktur dan permukaan kaca zink-tellurit. Satu siri sistem kaca tellurit ternari lutsinar dengan komposisi (80-*x*)TeO_2-20ZnO-*x*SiO_2, dengan x = 0 hingga 0.20 mol% telah disintesis melalui kaedah pelindapan peleburan konvensional. Kesan perubahan kandungan SiO_2 terhadap ketumpatan kaca, ciri struktur, sifat terma, sudut sentuh air (WCA), kekasaran permukaan dan morfologi permukaan telah dikaji. Sifat amorfus kaca telah disah menggunakan pengukuran pembelauan sinar-X. Peningkatan kandungan SiO_2 telah mengurangkan ketumpatan daripada 5.55 kepada 5.53 $g.cm^{-3}$ dan meningkatkan isipadu molar daripada 25.96 kepada 26.06 $cm^{-3}.mol^{-1}$. Analisis terma pembeza mendedahkan peningkatan suhu peralihan kaca (T_g) dengan peningkatan kandungan SiO_2. Faktor kestabilan terma didapati berubah antara 79.9 kepada 81.5°C bergantung kepada komposisi kaca. Morfologi sampel kaca telah diciri menggunakan mikroskopi daya atom dan pengukuran WCA. Kekasaran permukaan kaca sebanyak 10 nm telah dicapai iaitu lebih tinggi berbanding kaca biasa. Nilai optimum WCA yang telah diperolehi ialah 101° dan tenaga permukaan minimum yang didapati ialah 0.06 $N.m^{-1}$ bagi sampel kaca dengan 0.1 mol% SiO_2 menggambarkan kaca dengan kebolehbasahan yang rendah berbanding kaca zink-tellurit yang lain. Ciri struktur sampel kaca telah dikaji menggunakan spektroskopi inframerah transformasi Fourier (FTIR) dalam julat 600 – 4000 cm^{-1}. Keputusan FTIR menunjukkan koordinasi atom tellurium berubah daripada TeO_4 kepada TeO_3 dengan pertambahan kandungan SiO_2. Bagi koordinasi TeO_4, puncak jalur inframerah terjadi antara 642.8 dan 661.7 cm^{-1} sedangkan bagi TeO_3 pula, puncaknya telah dicerap antara 784.7 dan 773.1 cm^{-1}. Mikroskopi elektron pengimbasan pelepasan medan dan spektroskopi sinar-X tenaga tersebar telah dilaksanakan terhadap sampel kaca tersebut. Morfologi permukaan sistem kaca kelihatan lebih kasar dengan penambahan kandungan SiO_2 sehingga 0.1 mol% iaitu lebih baik berbanding kaca biasa. Secara keseluruhannya, sifat fizikal, terma dan struktur sistem kaca zink tellurit yang dicadangkan telah mengalami perubahan secara marginal manakala sifat permukaan telah bertambahbaik secara signifikan dengan penambahan SiO_2.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

n	-	Refractive index
n	-	Moles
P	-	Pressure
ρ	-	Density
r	-	Roughness of the surface
RMS	-	Root mean square roughness
R_a	-	Average roughness
R_p	-	Maximum peak
R_z	-	Mean height of roughness in 10 points
T	-	Temperature
T_m	-	Melting temperature
T_g	-	Glass transition temperature
T_c	-	Crystalline temperature
θ	-	Water contact angle
λ	-	Wavelength
γ_{lv}	-	Interfacial tension between liquid and vapour
γ_{sv}	-	Interfacial tension between solid and vapour
γ_{sl}	-	Interfacial tension between solid and liquid
δA	-	Total surface area
δG	-	Total surface free energy
θ_W	-	Wenzel's contact angle
θ_Y	-	Young's contact angle

LIST OF ABBREVIATIONS

AFM	-	Atomic Force Microscopy
BO	-	Bridging Oxygen
EDX	-	Energy Dispersive X-ray
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infrared
IR	-	Infrared
NBO	-	Non-bridging Oxygen
NPs	-	Nanoparticles
RMS	-	Root Mean Square
Tbp	-	Trigonal Bipyramid
Tp	-	Trigonal Pyramid
WCA		Water Contact Angle
XRD	-	X-ray Diffraction

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Introduction

In this chapter, the general information about this study will be provided in details. This study is about hydrophobic effect on tellurite glass with variation of dopant and modifier. The research background, problem statement, objective of the study, scope of study, significance of study and outline of study will be described in this chapter.

1.2 Background of the Study

In general, glass is a versatile and sometimes very enigmatic substance. It can be made stronger than steel or soluble in water, a detector of nuclear radiations or the source of a powerful laser beam depending upon composition (Pye, 1972). Glass is an amorphous solid or non-crystalline solids in which the atoms and molecules are not organized in periodic lattice pattern. The atomic arrangement of amorphous solids is no long range order but short or medium range order. In contrast, atomic arrangement in crystalline solids exhibit a property called long range order or translational periodicity which is the positions repeat in space in a regular array. The amorphous can be formed by fast cooling from a melt in order to avoid crystallization.

Telluriun oxide (TeO_2) based glasses has been scientific and technological interest due to their unique properties such as chemical durability, electrical conductivity, transmission capability, high dielectric constant, high refractive indices and low melting points (Nasu *et al*., 1990; Tanaba *et al*., 1990). Stanworth, (1952) and Burger *et al*., (1985) stated that the application of tellurite glasses in industries such as electric, optical, electronic and other fields are immense due to their good semiconducting properties. Recently, tellurite glasses have gained wide attention because of their potential as hosts of rare earth elements for the development of fibres and lasers covering all the main telecommunication bands (Conti *et al*., 2004), and promising materials for optical switching devices (Sidkey and Gaafar, 2004).

Besides that, tellurite glasses doped with heavy metal oxides or rare earth oxides (El-Mallawany *et al*., 1995; El-Mallawany, 1998; Berthereau *et al*., 1996) such as Nb_2O_3, CeO_2 or ZnO recently have received great scientific interest because these oxides can change the optical and physical properties of the tellurite glasses. Furthermore, ZnO–TeO_2 system is another basic system that has good glass forming ability and used by many researchers. Sidebottom *et al*., (1997) has reported that Zinc-tellurite glasses to be a suitable host for optically active rare earth ions because of the wide glass-formation range which is close to the extremum for binary tellurite glasses. In addition, ZnO–TeO_2 system was used as a basis for multicomponent optical glass synthesis and has been reported as a useful medium for ultralow loss (1 dB 1000 m^{-1}) optical fibers for wavelengths in the 3.5–4 µm region (Van Uitert and Wemple, 1978).

Therefore, from the coverage above it seems clear that tellurite glasses are strategically important solid materials. However, to my knowledge, using SiO_2 as one of the dopant in zinc-tellurite system is never been explored and documented. The hypersensitive transition of silicon ions manifest as anomalous sensitivity of line strength to the character of the dopant environment. Apart from their applications, there is a lack of data on structural investigation as well as the thermal properties of the zinc-tellurite glass system especially with addition of SiO_2 in the literature. Therefore, the aim of this research is to study the influence of SiO_2 on structural, thermal and surface texture properties of Zinc-tellurite glass system in order to understand the fundamental origin of such properties.

1.3 Problem Statement

The combination of tellurium oxide and zinc oxide forms stables glass system (Kozhukharov *et al*., 1986). Zinc-tellurite glass system is widely used in lasers, lens, optical and solar applications including solar power plants and solar collectors. The intensity of the incident sunlight determines the power of conversion of solar light to electricity or water heating. However, the glass material used results in reflection losses though convection and radiation losses are minimized. One of the important properties in self-cleaning glasses is refractive index (n). In order to make the surfaces anti-reflective for solar cover glasses, a low refractive index such as SiO_2 which $n \leq 1.4$ is needed (Surekha and Sudararajan, 2015).

In addition, SiO_2 doped zinc-tellurite glass are expectedly last longer than silica thin films since the substrate do not need to recoating after a long period of time because of few factors such as air, water and sunlight. Other than that, the light can passed through the glass easily compared to silicate thin films since the light can

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APPENDIX A

Pictures of Instruments Used

Figure A.1 : Density meter by Mettler Toledo available at Department of Physics, Faculty of Science, UTM.

Figure A.2 : Rigaku SmartLab X-ray diffractometer available at University Industry Research Laboratory, UTM.

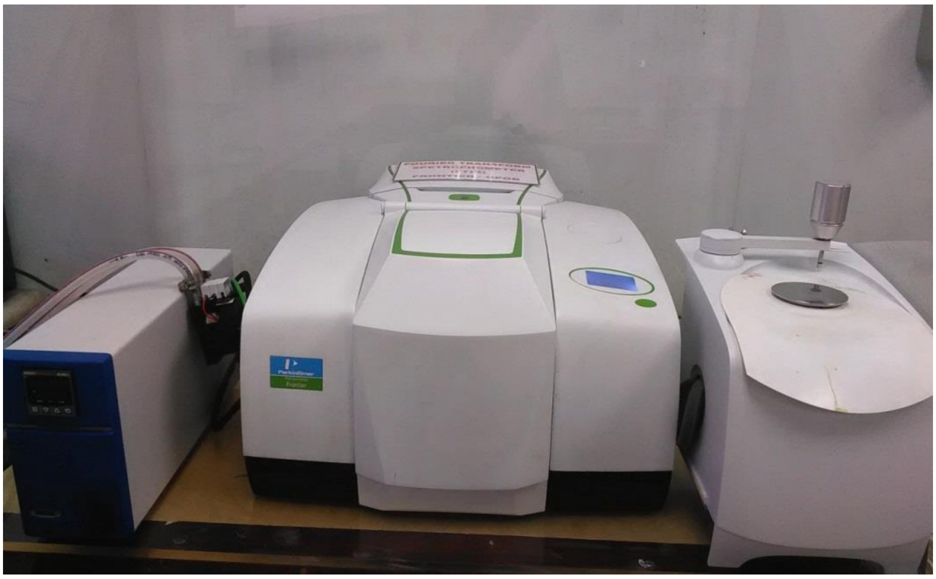


Figure A.3 : Perkin Elmer FTIR Spectrometer Frontier GP0B available at Chemistry Department, Faculty of Science, UTM.

Figure A.4 : Perkin Elmer STA 8000 available at University Industry Research Laboratory, UTM.

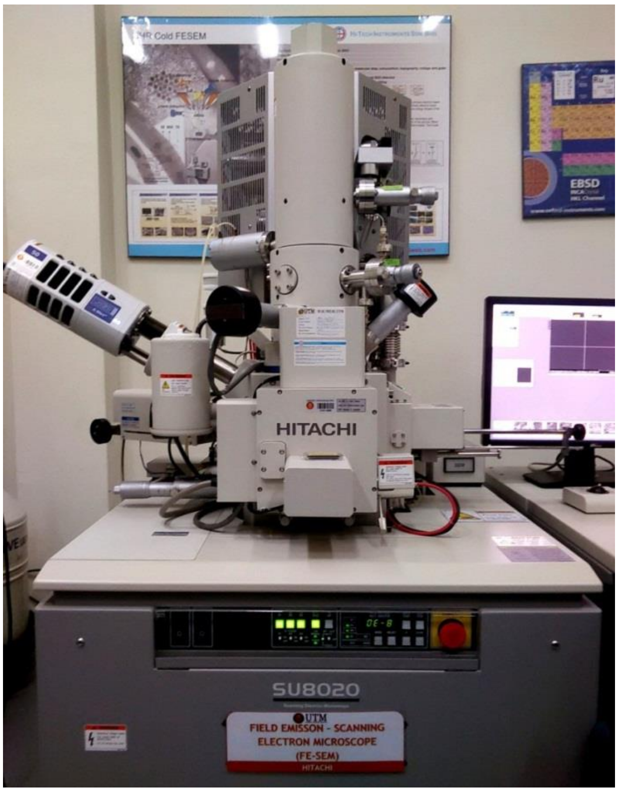


Figure A.5 : The hitachi SU8020 FESEM with EDX analysis available at University Industry Research Laboratory, UTM.

Figure A.6 : Atomic Force Microscope at Department of Physics, Faculty of Science, UTM.

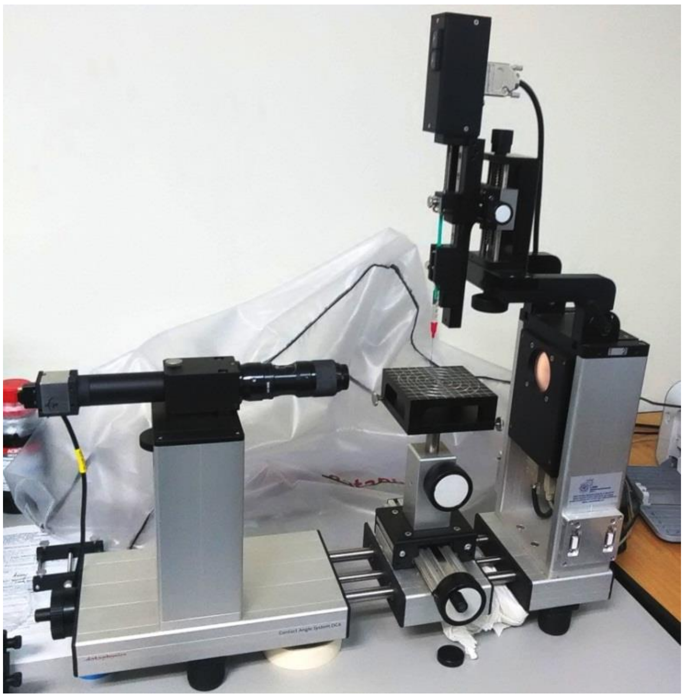


Figure A.7 : OCA meter by Data Physics available in Advance Membrane Technology Research Centre (AMTEC), UTM.

APPENDIX B

Calculation of Batch Sample Composition

Weight of sample = 15 g

Molar mass of each material:

- TeO_2 = 159.5988 g/mol
- ZnO = 81.3794 g/mol
- SiO_2 = 60.0843 g/mol

Example of mass determination for glass composition of (80-*x*) TeO_2 – 20ZnO – (*x*) SiO_2 system (*x* = 0.00, 0.05, 0.10, 0.15 and 0.20)

SAMPLE 1

S1: 80TeO_2 – 20ZnO

Total mol: (0.80 × 159.5988) + (0.20 × 81.3794) = 143.9549 g/mol

For 15 grams batch of glass:

$$TeO_2 = \frac{(0.80 \times 159.5988)}{143.9549} \times 15\text{ g} = 13.3041\text{ g}$$

ZnO = $\frac{(0.20\times 81.3794)}{143.9549}$ × 15 g = 1.6959 g

Total weight = 13.3041 + 1.6959

= 15 g.

<u>SAMPLE 2</u>

S2: 79.95 TeO_2 – 20ZnO – 0.05SiO

Total mol: (0.7995 × 159.5988) + (0.20 × 81.3794) + (0.0005 × 60.0843) = 143.9050 g/mol

For 15 grams batch of glass:

TeO_2 = $\frac{(0.7995\times 159.5988)}{143.9050}$ × 15 g = 13.3003 g

ZnO = $\frac{(0.20\times 81.3794)}{143.9050}$ × 15 g = 1.6965 g

SiO_2 = $\frac{(0.0005\times 60.0843)}{143.9050}$ × 15 g = 0.0031 g

Total weight = 13.3003 + 1.6965 + 0.0031

= 15 g.

Calculation for other samples (S3, S4 and S5) are the same.

APPENDIX C

Calculation for Density of Sample

$$\rho_s = \frac{w}{w-w'}\,\rho_o$$

where ρ_s is density of sample, w is weight in air, w' is weight in distilled water and ρ_o is density of distilled water.

<u>Sample 1</u>

Weight in air = 7.792 g

Weight in distilled water = 6.387 g

Density of distilled water = 1 g/cm^3

$$\rho_{s1} = \frac{7.792}{(7.792-6.387)}\ (1) = 5.546\ \text{g/cm}^3$$

Calculation for other samples (S2, S3, S4 and S5) are the same.

Calculation for Uncertainty of Density

$$\Delta\rho_s = \left(\frac{\Delta w}{w} + \frac{\Delta(w-w')}{(w-w')} \right) \rho_s$$

Where $\Delta\rho_s$ is uncertainty of sample density while ρ_s is density of sample, w is weight in air, w' is weight in distilled water, uncertainty of weight in air, Δw = 0.001 and Δ(w-w') = 0.002

Sample 1

ρ_{s1} = 5.546 g/mol

w = 7.792 g

w' = 6.387 g

Δw = 0.001

Δ(w-w') = 0.002

$$\Delta\rho_{s1} = \left(\frac{0.001}{7.792} + \frac{0.002}{1.405} \right) \times 5.546 = 0.009$$

Calculation for uncertainty of density for S2, S3, S4 and S5 are the same.

Calculation for Molar Volume of Sample

$$V_m = \frac{m}{\rho_s}$$

Where V_m is molar volume, ρ_s is density of sample and *m* is molecular weight.

Sample 1

Molecular weight for S1 = 143.9549 g/mol

Density of S1 = 5.546 g/cm^3

$$V_m = \frac{143.9549}{5.546} = 25.957 \text{ cm}^3\text{/mol}$$

Calculation for other samples (S2, S3, S4 and S5) are the same.

Calculation for Uncertainty of Molar Volume

$$\Delta V_m = \left(\frac{\Delta m}{m} + \frac{\Delta \rho_s}{\rho_s}\right) V_m$$

Where ΔV_m is uncertainty of molar volume, V_m is molar volume of sample, $\Delta\rho_s$ is uncertainty of sample density while ρ_s is density of sample, Δm = 0.001 and *m* is molecular weight of sample

Sample 1

ρ_{s1} = 5.546 g/mol

$\Delta\rho_{s1}$ = 0.0086

Δm = 0.001

m = 143.9549 g/mol

V_m = 25.957 cm^3/mol

$$\Delta V_m = \left(\frac{0.001}{143.9549} + \frac{0.0086}{5.546}\right) \text{ x } 25.957 = 0.0404$$

Calculation for uncertainty of molar volume for S2, S3, S4 and S5 are the same.

APPENDIX D

Calculation for Surface Tension

Young-Dupre´ Equation

$W_a = \gamma_L(1 + \cos\theta)$

Sample 1

$\gamma_L = 0.073$ N/m

$\theta = 83.15°$

$W_a = \gamma_L(1 + \cos\theta)$

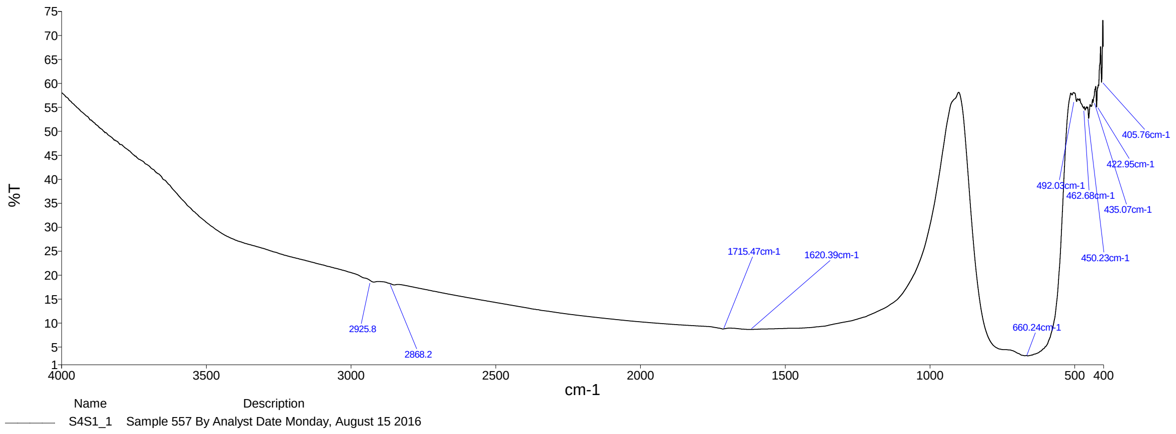
$W_a = 0.073(1 + \cos 83.15)$

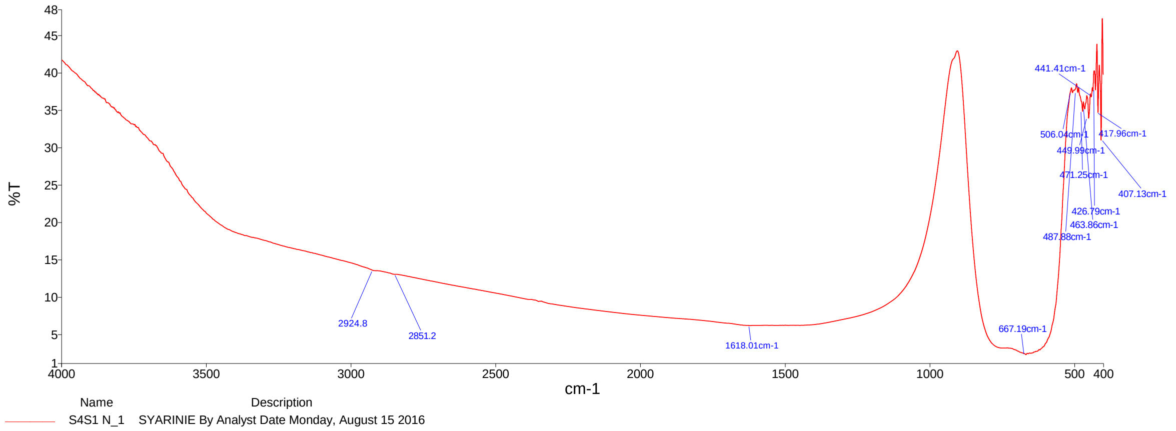
= 0.082 N/m

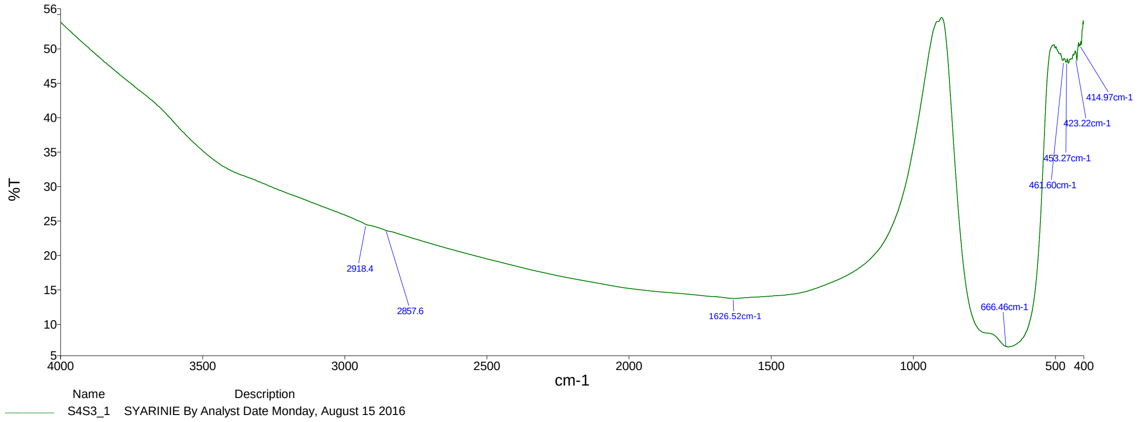
Calculation for surface tension sample S2, S3, S4 and S5 are the same.

APPENDIX E

FTIR SPECTRA



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666.46cm-1
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414.97cm-1
Name Description
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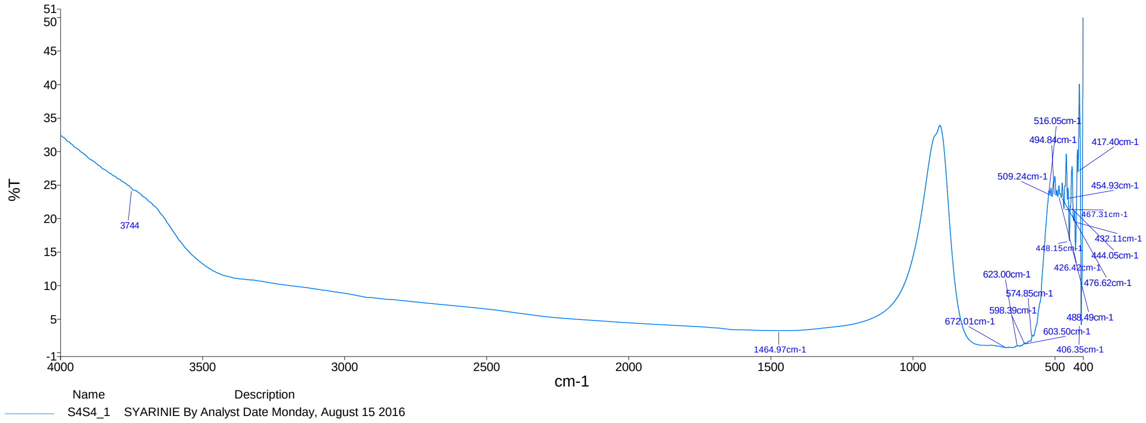
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432.11cm-1
448.15cm-1
444.05cm-1
426.42cm-1
476.62cm-1
623.00cm-1
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598.39cm-1
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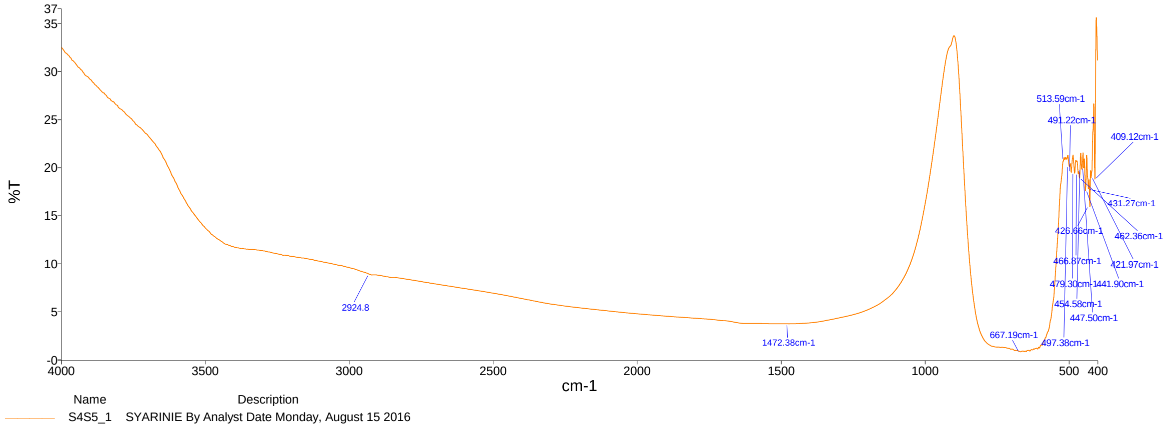
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441.90cm-1
454.58cm-1
447.50cm-1
667.19cm-1
497.38cm-1
Name Description
S4S5_1 SYARINIE By Analyst Date Monday, August 15 2016

APPENDIX F

List of Publications

Proceeding and Conference:

1. Azmi, S., Arifin, R. and Ghoshal, S. K. *Calcium Fluoride Concentration Effects on Themal and Hydrophobic Properties of Zinc-Tellurite Glass*. 4th International Science Postgraduate Conference 2016 (ISPC 2016) at Ibnu Sina Institute, Universiti Teknologi Malaysia (UTM), Johor Bahru, Johor on 22-24 February 2016.

2. Azmi, S., Arifin, R. and Ghoshal, S. K. *Improved Hydrophobicity of Silicon Dioxide Integrated Zinc-Tellurite Glass Surface*. 29th Regional Conference on Solid State Science and Technology 2016 (RCSSST 2016) at KSL Hotel, Johor Bahru, Johor on 15-17 November 2016.

3. Azmi, S., Arifin, R. and Ghoshal, S. K. (2017). Improved Hydrophobicity of Silicon Dioxide Integrated Zinc-Tellurite Glass Surface. *Solid State Phenomena*, 268, 87-91.