INFLUENCE OF BIMETALLIC TITANIUM AND SILVER NANOPARTICLES ON PHYSICAL AND SELF–CLEANING PROPERTIES OF ZINC–SILICATE–TELLURITE GLASS

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DEDICATION

To myself,
for the discipline and commitment that
I have put through this journey.

To my beloved parents (Abuya and Umi),
Siblings (Fadhil, Firdaus, Hariz, Akmal and Razin),
Sisters–in–law (Mie and Ika),
Acik's babies (Aniq, Afrina, Annur, Zahra and adik Annur).
For their endless love and supports

To my Eduwis family,
My coaches Mr Jinn and Mr Lee
My sisters Jia Xin and Saza
For crossing your path with me

To religion, nation and country.
Thank you

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ABSTRACT

In this study, the influence of two types of metallic (bimetallic) nanoparticles (NPs) including the pure silver (Ag) and titania (TiO_2) on the feasibility of improving the self-cleaning properties of some silicate zinc tellurite glasses were determined. To achieve this goal, the best from 3 series of glasses with the composition of $(79.93-\boldsymbol{z})$ $TeO_2 + 20ZnO + 0.06\ SiO_2 + 0.01TiO_2 + \boldsymbol{z}$ Ag, where $(0.01 \leq \boldsymbol{z} \leq 0.05$ mol%) were prepared via the standard melt-quenching method. As-quenched samples were characterized using diverse analytical measurements. The role of the varying Ag NPs and TiO_2 NPs (fixed content) on the hydrophobic and hydrophilic properties of the proposed glasses was evaluated. Highly transparent samples were obtained. The physical properties of the glasses such as the density, molar volume, theoretical crystalline volume, ionic and oxygen packing density of the best sample (S13) corresponded to 6.632 gcm^{-3}, 21.716 $cm^3\ mol^{-1}$, 12.729 $cm^3\ mol^{-1}$, 0.586 and 82.946 $molL^{-1}$, respectively. The surface plasmon resonance (SPR) absorption bands of these NPs were probed using the UV-Vis-NIR absorption spectroscopy. The X-ray diffraction (XRD) patterns verified the amorphous nature of the as-quenched samples. The energy dispersive X-ray (EDX) spectral analyses revealed the presence of the right elements in the composition. The scanning tunneling microscopy (STM) images and selected area electron diffraction (SAED) patterns confirmed the existence of the Ag and TiO_2 NPs inside the glass matrix. The measured thermal parameters (the glass transition, crystallization, and melting temperatures) of the samples obtained using the differential thermal analyzer (DTA) exhibited their good thermal stability over a wide glass formation region. The recorded Fourier transform infrared (FTIR) spectra of the glasses were complemented via the Raman analysis. The mechanical properties of the studied glasses including the Vickers hardness, fracture toughness, and brittleness were calculated which showed optimum values of 3268.08 MPa, 4.794 MPa $mm^{1/2}$, and 681.76 MPa $mm^{1/2}$, respectively for the S13 sample. The surface structure, texture, and morphology of the samples were evaluated using the field-emission scanning electron microscopy (FESEM), atomic force microscopy (AFM) and water contact angle (WCA) measurements. The experimental results on the self-cleaning traits (WCA and surface tension) of the glasses were compared with the theoretical calculation using the Young, Young-Dupre, Wenzel and Cassie-Baxter models. The sample S3 disclosed hydrophobic nature (with Young WCA of 112.39°) and the sample S6 displayed hydrophilic nature (with Young WCA of 86.27°) when included with TiO_2 NPs. It is affirmed that by manipulating the Ag NPs and TeO_2 concentrations in the proposed glasses, an improved self–cleaning properties can be achieved. The S13 sample showed the optimum hydrophobic traits with normalized roughness of 0.733 Nm; WCA of Young 97.47°, Wenzel 95.47°, Cassie-Baxter 130.13°. The optimal surface tension for the Young, Wenzel and surface energy of Young-Dupre for the S13 sample corresponded to 0.1727 Nm^{-1}, 0.1761 $Nm^{-1,}$ and 0.0626 Nm^{-1}, respectively. The results were analyzed, interpreted, compared and discussed. The mechanism behind the nanoparticles inclusion in assisting the improvement of the self-cleaning characteristics was understood.

ABSTRAK

Dalam kajian ini, pengaruh dua jenis nanozarah logam (dwilogam) termasuk perak tulen (Ag) dan titania (TiO_2) ke atas kemungkinan untuk memperbaiki sifat pembersihan-diri bagi beberapa kaca silika zink tellurit telah ditentukan. Untuk mencapai matlamat ini, sampel terbaik dari tiga siri kaca dengan komposisi $(79.93-\boldsymbol{z})$ $TeO_2 + 20ZnO + 0.06\ SiO_2 + 0.01TiO_2 + \boldsymbol{z}$ Ag, dimana $(0.01 \leq \boldsymbol{z} \leq 0.05$ mol%) telah disediakan dengan menggunakan kaedah lindap-kejut leburan. Sampel yang dilindap-kejut telah dicirikan menggunakan pelbagai pengukuran analitik. Peranan perubahan nanozarah Ag dan TiO_2 (kandungan tetap) pada sifat hidrofobik dan hidrofilik kaca yang diusulkan telah dinilai. Sampel yang tersangat lutsinar telah diperolehi. Sifat fizikal kaca seperti ketumpatan, isipadu molar, isipadu teori hablur, ketumpatan pemadatan ion dan oksigen bagi sampel terbaik (S13) berpadanan dengan to 6.632 gcm^{-3}, 21.716 $cm^3\ mol^{-1}$, 12.729 $cm^3\ mol^{-1}$, 0.586 dan 82.946 $molL^{-1}$ masing-masing. Jalur penyerapan resonans permukaan plasma bagi nanozarah ini disiasat menggunakan spektroskopi UV-Vis-NIR. Corak pembelauan sinar-X (XRD) mengesahkan keadaan amorfus sampel kaca terlindap-kejut. Analisis spektrum serakan tenaga sinar-X (EDX) mendedahkan kehadiran unsur yang betul dalam komposisi. Imej mikroskopi pengimbasan penerowongan (STM) dan corak pembelauan elektron kawasan terpilih mengesahkan kehadiran nanozarah perak dan titania di dalam matrik kaca. Parameter terma (suhu transisi kaca, suhu penghabluran dan suhu peleburan) bagi sampel yang diukur telah diperoleh menggunakan penganalisa terma pembeza (DTA) mempamerkan kestabilan terma yang baik bagi julat pembentukan kaca yang luas. Spektrum inframerah transformasi Fourier (FTIR) kaca yang direkodkan telah dilengkapkan oleh analisis Raman. Sifat-sifat mekanikal kaca yang dikaji termasuk kekerasan Vickers, keliatan patah and kerapuhan telah dikira yang mana menunjukkan nilai optimum masing-masing 3268.08 MPa, 4.794 MPa $mm^{1/2}$, dan 681.76 MPa $mm^{1/2}$, bagi sampel S13. Struktur permukaan, tekstur dan morfologi sampel telah dinilai menggunakan mikroskopi elektron pengimbasan-medan (FESEM), mikroskopi daya atomik (AFM) dan pengukuran sudut sentuh air (WCA). Keputusan eksperimen bagi ciri-ciri pembersihan-diri (WCA dan ketegangan permukaan) kaca dibandingkan dengan pengiraan teori dengan menggunakan model-model Young, Young-Dupre, Wenzel dan Cassie-Baxter. Sampel S3 mendedahkan sifat hidrofobik (dengan Young WCA 112.39°) dan sampel S6 mempamerkan sifat hidrofilik (dengan Young WCA 86.27°) apabila ditambah dengan nanozarah TiO_2. Adalah disahkan bahawa dengan memanipulasikan kandungan nanozarah Ag dan TeO_2 dalam kaca yang diusulkan, peningkatan ciri-ciri pembersiha- diri dapat dicapai. Sampel S13 menunjukkan ciri-ciri hidrofobik optimum dengan kekasaran normal 0.733 Nm, Young WCA 97.47°, Wenzel WCA 95.47°, Cassie-Baxter WCA 130.13°. Ketegangan permukaan optimal bagi Young, Wenzel dan tenaga permukaan Young-Dupre bagi sampel S13 sepadan masing-masing dengan 0.1727 Nm^{-1}, 0.1761 $Nm^{-1,}$ and 0.0626 Nm^{-1}. Semua keputusan telah dianalisa, ditafsir, dibanding dan dibincangkan. Mekanisma di sebalik penyertaan nanozarah dalam membantu peningkatan ciri-ciri pembersihan-diri telah difahami.

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LIST OF ABBREVIATIONS

AFM – Atomic Force Microscopy
Ag – Silver
Ag_2O – Silver Oxide
AgCl – Silver Chloride
Au – Gold
Al_2O_3 – Aluminium Oxide
AlF_3 – Aluminium Fluoride
B_2O_3 – Boron Oxide
Bi_2O_3 – Bismuth (III) Oxide
BO – Bridging Oxygen
BS – Beam Splitter
DTA – Differential Thermal Analysis
EDX – Energy Dispersive X–Ray Analysis
EM – Electromagnetic Radiation
Er_2O_3 – Erbium Oxide
FTIR – Fourier Transform Infrared Spectroscopy
Gd_2O_3 – Gadolinium (III) Oxide
HCl – Hydrochloric Acid
HF – Hydrogen Fluoride
HNO_3 – Nitric Acid
IFT – Interfacial Tension
IR – Infrared
JCPDS – Joint Committee on Powder Diffraction Standards
KBr – Potassium Bromide
L – Liquid
Li_2CO_3 – Lithium Carbonate
Li_2O – Lithium Oxide

MB – Methylene Blue

MgO – Magnesium Oxide

Na_2O – Sodium Oxide

Nb_2O_5 – Niobium Pentoxide

Nd_2O_3 – Neodymium Oxide

NPs – Nanoparticles

P_2O_5 – Phosphate Oxide

PbO – Lead Oxide

PDI – Perylene Diimide

Pt – Platinum

R&D – Research and Development

S – Solid

SAED – Selected Area Electron Diffraction

SE – Surface Energy

SEM – Scanning Electron Microscopy

SFM – Scanning Force Microscope

SiO_2 – Silicon Dioxide

Sm_2O_3 – Samarium (III) Oxide

SNOM – Scanning Near Field Optical Microscope

SPMs – Scanning Probe Microscopes

SPR – Surface Plasmon Resonance

SR – Surface Roughness

ST – Surface Tension

STM – Scanning Tunneling Microscope

Tb_2O_3 – Terbium (III) Oxide

tbp – Trigonal Bipyramids

TeO_2 – Tellurite

TiO_2 – Titania

TM – Transition Metal

tp – Trigonal Pyramid

TZS	–	Tellurite Zinc Silicate
UV	–	Ultraviolet
V	–	Gas or Vapour
WCA	–	Water Contact Angle
WO_3	–	Tungsten Trioxide
XRD	–	X–Ray Diffraction Analysis
ZnO	–	Zinc Oxide

LIST OF SYMBOLS

ΔT – Thermal Stability

a, c – Crack Indentation

α – Absorption Coefficient

B – Brittleness

c – The Speed of Light

C – Number of Oxygens Per Formula Unit

d – Distance Between Atomic Layers in Crystal

D – Density of Air

f – The Force Constant of The Bond

f_1 – Fractional of Apparent Projected Surface Area in Contact with Liquid

f_2 Fractional of Real Projected Surface Area

H_R – Hruby Number

H_V – Vickers Hardness

K_{IC} – Fracture Toughness

λ – Wavelength of The Incident X–Ray Beam

M – Glass Molecular Weight

m_o – The Atomic Weights in Kg of Anion

m_r – The Atomic Weights in Kg of Cation

N_A – Avogadro Number in mol $^{-1}$

n – Integer

θ – Angle

θ_Y – Young WCA

θ_W – Wenzel WCA

θ_{CB} – Cassie Baxter WCA

ρ – Density

ρ_o – Density of Distilled Water

r – Homogeneous Rough Surface

rm – Ionic Radii of The Metal

ro – Ionic Radii of The Oxygen

T_c – Crystallization Temperature

T_g – Glass Transition Temperature

T_m – Melting Temperature

μ – The Reduced Mass of The Cation–Anion Molecule

V – Wave Number Per Centimeter

V_c – Theoretical Crystalline Volume

V_{OPD} – Oxygen Packing Density

V_i – Molar Volume of the ith Component

V_m – Molar Volume

$V_{t,}$ – Ionic Packing Density

w_A – Weight of Sample in Air

w_1 – Weight in Distilled Water

W_{SL} – Reversible Work of Adhesion

γ_{SV} – IFT of Solid–Vapour

γ_{LV} – IFT of Liquid–Vapour

γ_{SL} – IFT of Solid–Liquid

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CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter presents the background, problem statement, objectives, scope, significance of this research and thesis outline.

1.2 Background of Research

Currently, the tellurite–based glasses have gained attention from other researchers due to its interesting optical, electrical and magnetic properties (Rafaella et al., 2001; El–Mallawany et al., 2002; El–Mallawany et al.,2004; Aoxiang et al., 2009; El–Mallawany et al.,2010; Bahadur et al., 2010; Chillcce et al., 2011; Goncalo et al., 2013; Asmahani et al., 2013; Asmahani et al., 2014; Asmahani et al., 2015; Yusoff et al., 2015; Ismail et al., 2016; Yusof et al., 2017; Nurhafizah et al., 2017; Azmi et al., 2017; Al–Hadeethi et al., 2020; Al–Buriahi et al., 2020; Sayyed et al., 2020). Tellurite–based glasses are very noticeable because of their exclusive properties such as excellent transmission in visible as well as IR wavelength regions, good in mechanical strength and chemical durability also high in electrical conductivity (Jaba et al., 2000; Mohamad et al., 2006; Sidek et al., 2013). Moreover, these glasses also possess excellent physical properties such as higher refractive index (in the range 2.0–2.5), low cut–off phonon energy (~700 cm^{-1}) and low melting temperature (733 ºC) where these properties contribute to high possibility of stable glass–forming using the conventional melt–quenching method.

The presence of zinc oxide (ZnO) as a network modifier in the tellurite glass develops the opacity of the glass. The ZnO inlusion causes a decrease in the melting point and low rates of crystallization, chemical durability and the nonlinear refractive index of a medium. A binary zinc–tellurite system have a significant solubility of transition oxide elements and a wide range of glass–forming, as it is considered as one of great interest to glass technologies and applications (Al–Buriahi et al., 2020; Halimah et al., 2020). Previous researches revealed that the existence of zinc in tellurite glasses was stable and have established interest from different researchers widely (Burger et al., 1992; Rafaella et al., 2001; Dousti et al., 2013; Halimah et al., 2020; Oliveira et al., 2020; Sayyed et al., 2020; Al–Buriahi et al., 2020; Al–Hadeethi et al., 2020). Previously, Tafida et al. (2020) studied the physical, morphology, thermal and structural properties of the different compositions of tellurite as a glass network former and zinc as the network modifier. It was reported that the ZnO encouraged the decrease in the melting point and also increased the ability of the glass formed during the glass production process (Rafaella et al., 2001; Tafida et al., 2020).

At excitation and lasing wavelength, the silicate glasses are chemically durable, thermally stable and optically transparent. Such high viscosity glasses can be formed, cooled and annealed without crystallization.. Silicate glasses are the most widely used glasses for consumer. The ease of manufacture and excellent transparency of visible light, which makes them particularly useful in optical telecommunications, micro and optoelectronics, and in near–IR windows due to their low optical attenuation and good optical dispersion (Rafaella et al., 2001; Dousti et al., 2013; Luciana et al., 2011). It has been reported that the existence of the silica plays crucial factors which enhanced the hydrophobic self–cleaning properties. The hydrophobic properties of silica displayed a water–resistant properties because of its nanostructure and chemical properties. When silica is applied to a surface of a material, the silica particles adhere to the host material and preventing the liquids from permeating the rough texture.

Silica was used to treat other surfaces to become more hydrophobic, due to the morphology of the silica particles once they adhered to the host of the material.

The silica particles alters the surface of its host material, as a result in possesses a hydrophobic surface (Parvatty et al., 2007; Bouzid et al., 2008; Satish et al., 2010; Pradip et al., 2011; Wael et al., 2017; Azmi et al., 2017). Azmi et al. (2017) reported that glasses with a nominal composition of $(80–x)TeO_2–xSiO_2–20ZnO$ revealed the hydrophobic self–cleaning properties where x is in between 0 to 0.2 mol%. A thermally stable glass improved the surface roughness (SR) and water contact angle (WCA) as the SiO_2 content increased. The enhancement was attributed to the reduction of active groups on the glass surface, where the SiO_2 assisted the surface chemistry with low interfacial free energy favoured the formation of hydrogen–bonds in contact with the water. However, at higher SiO_2 concentration (more than 0.10 mol%), a rapid reduction in the SR and WCA was due to the surface saturation effects (Azmi et al., 2017).

The fascinating properties of self–cleaning is inspired by a natural phenomenon such as the water striders, butterfly wings, mosquito eyes, and the most popular Nelumbo or known as the lotus leaf. The capability of the self–cleaning is significant for nature to defends itself from the dirt, and contaminants or pollutants. The wettability property and surface roughness are the crucial reasons for micro dirt particles to be picking up by the water droplets, reducing the droplet's adhesion to the surface. The hydrophobicity and self–cleaning properties are also presented in other plants, such as Tropaeolum (nasturtium), Opuntia (prickly pear), Alchemilla, cane and some insect wings (Mingqian et al., 2016). Interestingly, the rough surface discovered on the lotus leaf was the mastoid structure with the waxiness that holds up the droplets and slides easily while taking up all the dirt.

Inspired by the nature of the self–cleaning, the self–cleaning technologies had been particularly interested among researchers in late 1980s. The self–cleaning properties were achieved by controlling the surface wettability. It is evaluated with the value of WCA between water and the surface (Ganbavle et al., 2011; Linda et al., 2011; Mridul et al., 2014; Mohamed et al., 2015; Yusof et al., 2017). This hierarchical surface structure induced the durable water repellency and inspired the advancement of the self–cleaning technologies and applications, including the self–cleaning skyscraper windows as well as commercial products such as the tiles, textiles, paint for traffic marking and buildings. Thus, the customization of the surface wettability by controlling the WCA, surface roughness (SR), interfacial tension (IFT) and surface energy (SE) are fundamentals to determine the self–cleaning mechanisms known as the hydrophilic (wettable with low WCA) and hydrophobic (less wettable with high WCA) actions (Ismail et al., 2016; Nurhafizah et al., 2017; Yusof et al., 2017; Azmi et al., 2017).

The water droplets appear flat on the hydrophilic surface. The water droplets become spherical on the hydrophobic surface with high WCA which allows it rolling off with dirt and impurities, thereby offer the cleaning (Toshiaki et al., 2004; Marco et al., 2010; Mahmoud et al., 2014; Cohen et al., 2015; Xiao et al., 2018). Previous research showed that the glasses containing photocatalytic titania (TiO_2) either have hydrophilic or hydrophobic surfaces. However, most of the TiO_2 materials showed the hydrophilic property. Hence, the hydrophilic self–cleaning was achieved when water at the glass surface formed a layer and utilized the sunlight to carry away the dust and other impurities on the surfaces (Yusof et al., 2015; Yusof et al., 2017; Nurhafizah et al., 2017).

Meanwhile, the hydrophobic surfaces were attained by controlling the surface roughness or applying a low surface energy. The hydrophobic surface is self–cleaned when water forms a spherical droplet and rolls off while carried away dust and dirt. High water surface tension allows the droplets to take an almost spherical shape, as a sphere has a limit surface area, and this shape requires a less solid–liquid surface energy.

The adhesion forces resulting in complete or partial wetting is either depending on the structure of the surface or the fluid tension of the water droplets. The hydrophobic water–repellent surface structures enable the contact area and the adhesion force between the surface and droplet to reduce significantly, which resulting in a self–cleaning process. It is concluded that the wettability plays a significant role in determining the self–cleaning properties, either hydrophobic or hydrophilic.

Generally, silicates are often referring as hydrophobic depending on whether the adsorption of liquid is higher or lower into the surface. To the extent which silicates act as a catalyst or dopant to achieve hydrophilic or hydrophobic. (Parvatty et al., 2007; Bouzid et al., 2008; Satish et al., 2010; Pradip et al., 2011; Wael et al., 2017). Deepa et al. (2016) reported that the degree of hydrophobicity to surface coating also reduced a bacterial attachment leading to antimicrobial nanoparticles (Deepa et al., 2016). Huang et al. (2020) reported the superhydrophobic coatings with satisfactory self–cleaning capabilities, excellent thermal stabilities, and good mechanical properties were successfully fabricated with a mixed of silica nanoparticles and polyacrylate solutions (Huang et al., 2020). Liang et al. (2020) reported the superhydrophobic surface with high transmittance and excellent weather resistance superhydrophobic for the glass covers of solar cells are designed.

Past researches revealed, most of the existed hydrophobic self–cleaning SiO_2–based materials were in the form of thin films with or without coating. Although the approaches to achieve the self–cleaning glass surfaces have seldom been reported (Azmi et al., 2017), it is known that self–cleaning surface was created by producing an extra roughness on the material or by modified roughening via the selection of appropriate functional materials with low free surface energy (Huang et al., 2020). In addition, it was reported that (Azmi et al., 2017) glass with nominal compositions of $(80–x)TeO_2–xSiO_2–20ZnO$ revealed the hydrophobic self–cleaning property where x is between 0.00 to 0.20 mol%. The highest optimum WCA reported was 101.02°, with 0.10 mol% of SiO_2. Therefore, a series of new zinc tellurite glass compositions with SiO_2 between 0.00 to 0.10 mol% are prepared to obtain the improve hydrophobic surfaces.

Bismarck et al. (2004) characterized the glass fibres obtained from silicate waste, and various commercial glass fibres revealed the hydrophobic where the WCA of the glass fibres against water and diiodomethane are closer to 90°. It stated that the high–energy surface tends to absorb the moisture and other contaminants in a standard atmosphere. It is also reported that the surface tension of the fibres was somewhat similar to a polymer surfaces. Li et al. (2008) reported the cotton fabrics treated with silica sol and hexadecyltrimethoxysilane (HDTMS) prepared by sol–gel method showed an excellent hydrophobic properties. The surfaces were obtained by dip–coating the silica hydrosols prepared via hydrolysis and condensation of water glass onto cotton substrates. The surface of the silica coating was modified with a non–fluoro compound HDTMS, gain a thin film through self–assembly, superhydrophobicity with a WCA higher than 151.28° was achieved. Azmi et al. (2017) reported the hydrophobic traits self–cleaning glass without coating consisted variation of silica into the zinc tellurite host glasses. The addition of silica improved surface roughness thus increased the value of WCA.

It is needless to say that the glass systems with the significant properties of extreme durability, thermal stability, and transparency have gained particular attention in the building and construction industries, especially in terms of self–cleaning materials. The use of this self–cleaning glass is somewhat uncommon. However, it would minimize both cleanliness maintenance time and cost, resulting in a more economical and environmentally friendly material (Ampornphan et al., 2014; Dorel et al., 2008). The self–cleaning glass systems were also seen as a revolutionary and the practical material for pollutant removal and energy production. The advanced research in self–cleaning discovery leads to many commercial applications including tiles, textiles, traffic marking paint and paint for buildings with self–cleaning properties (Haleh et al., 2016; Fei et al., 2017; Maryam et al., 2017). These coatings decrease the use of detergents, solvents, and water. It also saves large volumes of traditional paints, and more interestingly, the coatings protect the buildings from the UV degradation.

Extensive studies were performed to develop highly efficient and robust self–cleaning surfaces via coating which also improved the optical performance (Kazuhito et al., 2005; Kazuya et al., 2012; Yelda et al., 2010). The optical qualities such as high refractive index and excellent transmittance of incident light are essential for self–cleaning applications, especially in the optoelectronic device, microfluidic devices, biomedical science, ships, automotive, self–cleaning windows, buildings, self–cleaning oven and solar panel. The transition metal (TM) doped oxide glasses have wide applications in the field of compact lenses and switching devices, cathode materials in batteries and optoelectronic devices. Among various transition metal oxides, titania had been widely studied due to its unique properties such as large band gap, transparent to visible light, high refractive index and excellent chemical stability. The titanium ions participated in the glass network as Ti^{3+} and Ti^{4+}; hence the addition of TiO_2 resulted in the modification of physical and structural properties (Adriana et al., 2008; Kazuhito et al., 2005).

Various researchers showed that the glasses contained titania (TiO_2) either had photocatalytic and hydrophilic, (Kazuhito et al., 2005; Adriana et al., 2008; Dorel et al., 2008; Yelda et al., 2010, Kazuya et al., 2012; Mridul et al., 2014; Kundu et al., 2014; Berwal et al., 2015; Ismail et al., 2016) hydrophilic and oleophilic surfaces in the same time, (Wang et al., 1997) also reported hydrophobic (Alfa et al., 2019; Rosales et al., 2020). This changeability results concluded that the addition of TiO_2 into the system might make the surfaces behaves and hydrophilic or hydrophobic depends on the nature of the system itself. Furthermore, it was demonstrated that the hydrophilicity being a self–cleaning featured flat glass surfaces mitigated the harm caused by the dirt. The TiO_2 thin films coated glass surface were used for self–cleaning, anti–fog, anti–bacterial and anti–pollution applications. Yusof et al. (2017) reported that glass with the composition $(69–x)TeO_2–20ZnO–10Na_2O–1Er_2O_3–xTiO_2$, where $x = 0.0, 0.1, 0.2, 0.3$ and 0.4 mol% revealed hydrophilic properties as the WCA decreased from 68° to 43° with increasing TiO_2 NPs concentration.

The inclusion of ZnO as a modifier improved the stability of the glass through the thermal characteristics. Ismail et al. (2016) reported the influence TiO_2 NPs on the structural and self–cleaning properties of the glasses with a nominal composition of $(42–x)P_2O_5–8MgO–50ZnO–xTiO_2$ with $x = 0, 1, 2, 3$ and 4 mol%. The WCA increased from 63.1° to 70.0° with increasing of TiO_2 NPs. The presence of ZnO in the glass system helps to stabilize the glass system by minimized the hygroscopic nature. The relationship between TiO_2 NPs assisted spectral modification, and self−cleaning ability was also reported. Nurhafizah et al. (2017) reported the effect of the embedment of AgCl on the structural and self–cleaning properties of the glass with the nominal composition of $(68-x)TeO_2–15Li_2CO_3–15Nb_2O_5–1Er_2O_3–1Nd_2O_3–xAgCl$ with x = 1.0, 2.0 and 3.0 mol%. The glasses revealed an increased value of WCA from 25.5° to 47.4° with increasing concentration of Ag NPs. The interplay between Ag NPs and TeO_2 leads to hydrophilic self–cleaning properties with photocatalytic action. Silver at the nanoscale is promising as an alternative water disinfectant because of its unique physicochemical properties and excellent antimicrobial action (Nurhafizah et al., 2017). In this previous research, glasses containing bimetallic NPs such as TiO_2 or Ag showed photocatalytic action. In fact, the dust and other impurities on the surface of the material are easily removed in the presence of sunlight, leading to hydrophilic self–cleaning properties (Ismail et al., 2016; Yusof et al., 2017; Nurhafizah et al., 2017).

Only a few successful researches reported the hydrophilic self–cleaning (Ismail et al., 2016; Yusof et al., 2017; Nurhafizah et al., 2017) and hydrophobic self–cleaning (Azmi et al., 2017) glasses without coating. Most of the previous research reported that hydrophobic self–cleaning was from thin films, glass fibres, biomaterials and mostly achieved via surface coating (Bismarck et al., 2004; Parvatty et al., 2007; Bouzid et al., 2008; Li et al., 2008; Satish et al., 2010; Pradip et al., 2011; Deepa et al., 2016; Wael et al., 2017; Huang et al., 2020). Bouzid et al. (2008) reported that the silica–derived biomaterials self–cleaning were achieved by selecting the appropriate surface chemistry. The biocompatibility may depend on the proper balance of hydrophilic and hydrophobic groups. Satish et al. (2010) also reported the transparent superhydrophobic coating had excellent wetting behaviour properties with high optical transmission, thermal stability and imperviousness against strong acids by controlling the SR of resultant coatings. Deepa et al. (2016) reported by controlling the surface morphology and properties of the silica nanoparticles, the superhydrophobic were achieved for thin films of perylene diimide (PDI) via coating.

However, none of these reported the correlation between the wettability, SR and IFT, including the theoretical calculation related to Young Dupree, Wenzel, and Cassie–Baxter, especially for glass without coating. Consequently, this research resolves to provide the correlation between the morphology, physical, thermal, structural, mechanical and surface with the self–cleaning properties of wettability, SR via AFM, including the theoretical calculation of IFT and WCA related to Young, Young Dupree, Wenzel, and Cassie–Baxter models.

1.3 Problem Statement

Among the oxides glasses, high demand of tellurium oxide (TeO_2) host glass gained significant attention from several researchers due to its attractive properties for many technologic purposes (Rafaella et al., 2001; Aoxiang et al., 2009; Bahadur et al., 2010; Goncalo et al., 2013; El–Mallawany, 2002; El–Mallawany et al., 2004; El–Mallawany et al., 2010; Al–Hadeethi et al., 2020; Al–Buriahi et al., 2020; Sayyed et al. 2020). Glasses with primary network former of TeO_2 offer a continuous glass–forming region, yielding attractive and stable. The glass can easily form by adding a suitable modifier such as zinc oxide (ZnO).

The presence of ZnO as a network modifier in the tellurite glass develops the solubility of transition oxide elements and a wide range of glass–forming, which it is considered as one of great interest to glass technologies and applications. Earlier reports in the literature showed that the studies on the self–cleaning traits of the glasses had been deficient. A careful examination and basic understanding of the self–cleaning properties of the glass can lead to many technological advances. Based on these facts, this research intends to gain a deep insight into the self–cleaning properties of the zinc tellurite glass systems.

The photocatalytic activity was induced on the glass surface through the coating procedure promoted the self–cleaning action (Dorel et al., 2008; Kazuya et al., 2012; Maryam et al., 2017). However, the leaching problem which associated with the coating materials that purged from the glass surface makes it impotent to maintain the elongated self–cleanliness of the glass system. Another successful self–cleaning glass without coating was prepared to achieve the hydrophilic (Yusof et al., 2015; Yusof et al., 2017; Nurhafizah et al., 2017) and hydrophobic properties (Azmi et al., 2017). It is reported that the influence of TiO_2 NPs inclusion into the glass system (Yusof et al., 2017; Ismail et al., .2016) displayed an opposite trend where Yusof et al. (2017) showed a decrease in the WCA values while Ismail et al. (2016) exhibited an increase in the WCA value with the rise in the concentration of TiO_2 NPs.

However, both types of research revealed that the glass is in hydrophilic self–cleaning state as the WCA reported is less than 90. Yusof et al. (2017) reported 0.00 to 0.40 mol% and Ismail et al. (2016) reported 0 to 4 mol% of the TiO_2 NPs to obtained the hydrophilic self–cleaning glass. This present research uses mol% of SiO_2 in between 0.00 to 0.10 mol%; therefore, the mol% of NPs cannot be more significant than the SiO_2. Therefore, the TiO_2 NPs are varying in between 0.00 to 0.05 mol%. The combination material of hydrophobic (SiO_2) and hydrophilic (TiO_2 NPs) in this present research, and it is expected that the smallest values of TiO_2 NPs transit the glass state from hydrophobic into hydrophilic based on previous research due to its stronger photocatalytic properties.

In this research, the Ag NPs are varying in between 0.00 to 0.05 mol% same as TiO_2 NPs. Despite all of these efforts, the correlation between the ST and WCA and the further understanding based on the Young, Young–Dupre, Wenzel and Cassie–Baxter theories of the self–cleaning glasses have been deficient. Hence, it is essential to understand the basic mechanism of the self–cleaning (hydrophilic and hydrophobic) and calculate the relevant parameters relates to the Young, Young–Dupre, Wenzel, and Cassie–Baxter models for clarifying the self–cleaning attributes.

Considering the immense fundamental and applied significance of the self–cleaning glasses, it is believed that a proper understanding of the self–cleaning mechanism in the glasses can improve the self–cleaning performance due to the co–embedment of two types of NPs (bimetallic) which is essential for future applications. The role of the bimetallic NPs activation on the hydrophobic (lotus leaf effect) properties of zinc–silicate tellurite glass system remains unclear and less documented. Moreover, the correlation between WCA, wettability and roughness for glasses are lacking in documentation and knowledge. Furthermore, the basic mechanism behind the self–cleaning, including theoretical calculation of Young, Young Dupre, Wenzel and Cassie–Baxter is not yet to be discussed thoroughly. Therefore, the zinc–silicate tellurite glasses with bimetallic TiO_2 and Ag NPs are prepared and characterize to resolve the self–cleaning drawback abilities of the transition from hydrophilic to hydrophobic. Previous research revealed that each metallic NPs showed high compatibility between Ag and TiO_2 which is crucial for this research (Ismail et al., 2016; Fei et al., 2017; Nurhafizah et al., 2017; Wael et al., 2017), however, none of the researches reported the embedment of bimetallic NPs in one glass system.

To this date, the zinc–silicate tellurite glass system embedded with titania and silver nanoparticles has not been studied to determine their self–cleaning performance. Thus, the careful synthesis and details characterizations of these glasses are necessary to determine the improvement of the self–cleaning water repellent (hydrophobic) traits due to the activation of two types of nanoparticles. Therefore, the primary aim of this study is to investigate the impact of bimetallic titania (TiO_2) and silver (Ag) NPs on self–cleanliness properties on zinc–silicate tellurite glass. The combination of TiO_2 and Ag NPs are expected to improve the hydrophobic self–cleanliness property of SiO_2. These compositions are not yet reported in any research, thus making it the first hydrophobic self–cleaning glasses without coating with the embedment of bimetallic NPs. Additionally, the morphology, physical, thermal, structural, mechanical, and surface were carried out to determine the correlation with self–cleaning properties of the prepared samples. The physics behind the origin of self–cleaning property (hydrophobic and hydrophilic) will be fully understood by comparing the experimental data with the theoretical model of Young, Young Dupree, Wenzel, and Cassie–Baxter.

1.4 Objectives of Research

i. To synthesis three series of bimetallic nanoparticles of titania and silver activated hydrophobic zinc–silicate tellurite glass system via melt–quenching technique with nominal composition of $(80–\boldsymbol{x})TeO_2–20ZnO–\boldsymbol{x}SiO_2$, $(79.94–\mathbf{y})TeO_2–20ZnO–0.06SiO_2–\boldsymbol{y}TiO_2$ and $(79.93–\mathbf{z})TeO_2–20ZnO–0.06SiO_2–0.01TiO_2–\mathbf{z}Ag$ respectively with Series 1, 2 and 3.

ii. To determine the influence of bimetallic nanoparticles activation on the physical, thermal, structural, mechanical and surface properties of zinc silicate tellurite glasses.

iii. To evaluate the effects of silicon dioxide and metal nanoparticles concentration on the improvement of hydrophobic activity of the glass surface in terms of water contact angle and wettability.

iv. To compare the experimental data with theoretical estimates from Young, Young–Dupre, Wenzel, and Cassie–Baxter model calculation for understanding the self–cleaning mechanism.

1.5 Scope of the Research

This research consists three series of zinc silicate tellurite glass embedded with the bimetallic NPs, TiO_2 and Ag via melt–quenching technique. The first series of glass highlights the effect of dopant SiO_2 on host glasses with nominal composition $(80–x)TeO_2–20ZnO–xSiO_2$ (where x are varied as 0.00, 0.03, 0.06, 0.09, and 0.12 mol%) are prepared without any NPs embedment. By taking the optimum composition from the first series, the second series of glass highlights the embedment of metallic TiO_2 NPs with nominal compositions $(79.94–y)TeO_2–20ZnO–0.06SiO_2–yTiO_2$ NPs (where y are varied as 0.00, 0.01, 0.02, 0.03, 0.04, and 0.05 mol%) are synthesize.

By choosing the optimum sample from the second series, the third series glasses with embedment of Ag NPs are prepare with nominal compositions (79.93–z)TeO_2–20ZnO–0.06 SiO_2–0.01TiO_2–zAg NPs (where z is varied as 0.00, 0.01, 0.02, 0.03, 0.04, and 0.05 mol%). The optimum glass from each series is evaluated and determine from the surface properties by WCA and AFM characterization.

The analysis consists of morphology physical, thermal, structural, mechanical and surface properties towards self–cleaning performance. The morphology properties are determined by X–ray Diffraction Analysis (XRD) and Energy Dispersive X–ray Analysis (EDX). The physical properties observed are density, molar and theoretical crystalline volume, the ionic and oxygen packing density of glass. The energy transfers between bimetallic NPs are presented by Surface Plasmon Resonance (SPR). The existence of TiO_2 and Ag NPs are confirmed by Scanning Tunneling Microscopy (STM), while the thermal properties, including glass stability, are determined by Differential Thermal Analysis (DTA). The structural properties of the glass are studied using Fourier Transform Infrared Spectroscopy (FTIR) and RAMAN Spectroscopy. The glass mechanical properties are also determined by using the Vickers Hardness with the theoretical calculation of fracture toughness and brittleness.

The self–cleaning properties are investigated by Atomic Force Microscopy (AFM) and Water Contact Angle (WCA). The experimental approach was completed with the theoretical calculations of WCA parameters, using Young, Young's Dupree, Wenzel, and Cassie–Baxter models. All research experiments provided are highly relevant for the applied technology for efficient self–cleaning glasses.

1.6 Significance of the Research

This research aims to prepare the glass with hydrophobic abilities and investigate its self–cleaning properties. The economically practicable, environmental amiable and maintenance–free glass surfaces with improving hydrophobic activity are exceedingly demanding for several industrial purposes. Pollutant and dirt depositions on the glass surface which cause the visual obscurity and damages of the cultural heritages need to be inhibited. This proposed that the glasses without coating can minimize the leaching problem associate with the coating materials that purge from the glass surface make it impotent to maintain the elongated self–cleanliness of the glass system.

So far, none of the previous research reported the self–cleaning glass embedded with bimetallic nanoparticles; therefore, this research prepares the first self–cleaning tellurite zinc silicate glasses embedded with bimetallic Ag and TiO_2 NPs via melt–quenching technique that able to tailor the self–cleaning properties of the glass. It is established that by controlling the contents of Ag NPs and the existence of TiO_2 NPs, the hydrophobic and hydrophilic traits can be tailor, thereby enabling the proposed glass compositions suitable for diverse self–cleaning applications. In this regard, the basic understanding of the mechanism of hydrophobic interactions assisted self–cleaning traits of glass is essential. Therefore, the proposed glass in this research gives a significant contribution to improve the applications of self–cleaning glass in optoelectronic devices, microfluidic devices, biomedical science, ships, automotive, self–cleaning windows, buildings, self–cleaning oven and solar panel.

The fundamental understanding of Young Dupre, Wenzel and Cassie–Baxter theories on the self–cleaning properties were proven by mathematical calculation through WCA and IFT. In conclusion, this research is vital to increase the understanding of self–cleaning mechanisms and enhance the country's fame through the discovery in the field of self–cleaning glass.

1.7 Thesis Outline

This thesis describes the preparation and characterization of zinc silicate tellurite embedded with bimetallic TiO_2 and Ag NPs glasses. This thesis is divided into five chapters.

Chapter 1 introduced the purpose of this research, including a background of research, problem statement, objectives of research, the scope of research, the significance of the research and thesis outline.

Chapter 2 discusses the theories of the glass including the glass formation, telluride dioxide as glass network former, zinc oxide as glass network modifier, silicon dioxide as glass dopant, and titania and silver nanoparticles as bimetallic system. The X–ray Diffraction of glasses, the physical properties of tellurite glass, the thermal parameters of glasses, the structural properties, including the Fourier Transform Infrared Spectroscopy (FTIR) and Raman Spectroscopy, Energy Dispersive X–ray (EDX) spectroscopy of glasses are described in details. The mechanical properties of Vickers hardness measurements, including theoretical of fracture toughness and brittleness, are determined and calculated. The surface properties are done via Atomic Force Microscope (AFM) and discussed in detail with the correlation with self–cleaning properties. The self–cleaning properties, including Water Contact Angle (WCA) and interfacial tension (IFT), are explained, including their correlation with the theoretical calculation of Young, Young Dupree, Wenzel, and Cassie–Baxter.

Chapter 3 describes the details of melt–quenching technique for glass preparation, which including sample preparation, materials. In this chapter, the optimum glasses in each series are explained in the composition and the nominal composition of the glass sample. The experimental procedures are explained; consist of the X–ray Diffractometer (XRD), Energy Dispersive X–ray (EDX), Differential Thermal Analyzer (DTA), Fourier Transform Infrared Spectrometer (FTIR), Raman

Spectrometer, Vickers Hardness, Atomic Force Microscopy (AFM) and Water Contact Angle (WCA) Measurement.

Chapter 4 presents and discusses the results of this present research. The glass composition and formation for Series 1, 2 and 3 are discussed. The amorphous state of glass is proven by the X–ray Diffraction Analysis. The physical properties debated the correlation between the density, molar and theoretical crystalline volume, ionic and oxygen packing density. The thermal properties displayed the typical DTA curve with thermal parameters and the correlation with the physical properties. The analysis of the Fourier Transform Infrared (FTIR) and Raman are complemented each other to determine the structural properties. The elements of the material in this glass are displayed in the energy dispersive X–ray (EDX) spectrum. The existence of bimetallic NPs is evidenced by surface plasmon resonance. The mechanical properties discussed including Vickers hardness, Fracture Toughness and Brittleness. The surface analysis, including surface roughness and normalized surface roughness, are explained via Atomic Force Microscopy (AFM). The relation between Young WCA and normalized surface roughness are discussed. The theoretical calculation of interfacial tension (IFT) and WCA from Young, Wenzel and Young Dupree model are discussed and compared.

Chapter 5 concludes the thesis by discussing the objectives of this research. This chapter consists of an introduction, conclusion and further outlook for further research.

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1. Nazhirah S.N.M., Ghoshal S.K., Arifin R., Hamzah K., (2020). Titania Nanoparticles Activated Tellurite Zinc–Silicate Glass with Controlled Hydrophobic and Hydrophilic Traits for Self–Cleaning Applications. *Journal of Non–Crystalline Solids 530*:119778. **(Q1, IF:2.929)**.

Indexed Journal

1. Nazhirah Mazlan S.N., Arifin R., Ghoshal S.K., (2017). Hardness and Structure of Er^{3+}: Sm^{3+} Co–Doped Oxychloride Zinc Tellurite Glass. *Solid State Phenomena 268:* 43–47. **(Indexed by SCOPUS)**.
2. Nazhirah Mazlan S.N., Arifin R., Ghoshal S.K., (2018). Hydrophobic Zinc–Tellurite Glass System as Self–Cleaning Vehicle: Interplay amid SiO_2 and TeO_2. *Malaysian Journal of Fundamental and Applied Sciences Special Issue on Natural Sciences and Mathematics*: 492–494. **(Indexed by WOS)**.