

Physical and structure properties of QS-P₂O₅-CaO-BaO-Gd₂O₃/GdF₃ glasses medium doped with europium (III)

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ABSTRACT

Optical glass media derived from native natural resources, specifically Huta Ginjang (HG) quartz sand, were synthesized using a melt-quenching technique at 1200°C. The chemical composition of the glass matrix adhered to the formulations 15QS + 57 P₂O₅ + 15 CaO + 5 BaO + 5 Gd₂O₃ + 3 Eu₂O₃ and 15QS + 57 P₂O₅ + 15 CaO + 5 BaO + 5 GdF₃ + 3 Eu₂O₃. Each sample was doped with active Europium (Eu³⁺) ions at a concentration of 3 mol%, thus creating a difference between the two samples based on the incorporation of Gd₂O₃ (PCBGO) with GdF₃ (PCBGEF). The formed samples were then cut and polished to achieve optimal dimensions and transparency. Various physical characteristics, including density, ion concentration, and overall molar volume, were assessed using Archimedes' principle in addition to numerical calculations. In contrast, the structural properties were evaluated through fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) techniques to elucidate the functional groups present in the structure and diffraction patterns exhibited by the samples. The results of this study were conducted to contribute to the advancement of silica-phosphate glass materials intended for optical medium amplifiers using rare earth ions.

Keywords: Europium; gadolinium; optical medium; quartz sand

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INTRODUCTION

Quartz sand is one of Indonesia's abundant natural resources, particularly in Huta Ginjang, North Sumatra. With its high silica (SiO₂) content, quartz sand has great potential for use as a base material for functional materials, one of which is as a host matrix for rare earth ions [1]. In addition to being inexpensive, silica oxide (SiO₂) has several advantages as a host matrix for rare earth ions, particularly Europium (Eu³⁺), in luminescent material applications, including high optical transparency, thermal and chemical stability, good doping ability, and compatibility with various synthesis methods [2, 3]. Silica can be processed through various synthesis methods, such as sol-gel, melting, and chemical vapor deposition, thus providing flexibility in the manufacture of Eu³⁺ based luminescent materials [4]. P₂O₅-CaO-BaO serves to increase

the thermal stability, density, and chemical resistance of materials, support radiation protection, and influence and facilitate the substitution of rare earth ions for the structural and optical properties of glass [5]. Previous studies have shown that the use of phosphate (P₂O₅) and silicate (SiO₂) glass systems as host matrix media for rare earths, particularly Eu³⁺, yields positive results, especially in improving the optical radiative efficiency in these media [6]. Eu³⁺ ions produce intense redlight emission under X-ray excitation, making them promising candidates for this application. Previous studies have shown that rare earth-based materials doped with Eu³⁺ have high luminescence efficiency. For example, research by Fang et al. (2023) showed that Eu³⁺ ions can increase X-ray sensitivity by up to 30% compared to undoped materials [7]. Other research revealed that the use of glass matrices with certain modifications can improve the thermal stability

and luminescence efficiency of materials [8]. However, research related to the use of quartz sand as a base material in the manufacture of host matrices for rare earth ions is still very limited. This study shows that the combination of phosphate compounds and quartz sand has high potential for producing glass matrices with superior optical properties [9]. This study discusses the use of local quartz sand from Huta Ginjang combined with glass-forming compounds and Eu^{3+} dopants. The difference between the types of Gadolinium, namely Gadolinium Oxide (Gd_2O_3) and Gadolinium Fluoride (GdF_3), shows a significant difference in optical properties (such as lenses or radiation shields and radiation detectors or scintillators) and structure (such as orthorhombic or cubic) [10]. This is the reason why researchers utilize and combine these two compounds in order to produce a glass medium with better radiative properties. However, limitations such as cost have led researchers to utilize Huta Ginjang quartz sand due to its very high SiO_2 content, which reaches 97% [11].

LITERATURE REVIEW

Quartz sand is known as a non-metallic natural resource with significant potential, which can be used for various industrial purposes, such as in the manufacture of glass, cement, ceramics, and electronics [12]. The high silica content in quartz sand, reaching 98.21%, as well as low metal content such as iron and aluminum, makes it a potential candidate for applications in optical media [13]. Optical media are materials or substances that are specifically designed to interact with light in various ways, such as by reflecting, emitting, modifying, or regulating light according to specific characteristics, such as intensity, polarization, wavelength, or phase [14]. Europium (Eu^{3+}) in doping is a rare earth element that has unique luminescence properties, making it widely used in various optical and lighting applications [15]. Fourier Transform Infra-Red (FTIR) testing was used to compare various species of amorphous silica,

short-structured silicate, and silicic acid. The FTIR technique identifies various silicon species based on their characteristic absorption bands, such as the Si–O–Si and Si–OH absorption bands [16]. Another test is X-ray Diffraction (XRD), which is a technique used to analyze the crystalline structure of a material by observing the X-ray diffraction pattern produced by the interaction of rays with atoms in the crystal lattice [17].

RESEARCH METHODS

The materials used include phosphate oxide (P_2O_5), silica oxide (SiO_2) from quartz sand (QS), calcium oxide (CaO), barium oxide (BaO), gadolinium oxide (Gd_2O_3), gadolinium fluoride (GdF_3), and europium oxide (Eu_2O_3) have a high purity level of $> 99\%$. All compounds were composed into two glass samples with the following chemical equations: $15\text{QS} - 57 \text{P}_2\text{O}_5 - 15 \text{CaO} - 5 \text{BaO} - 5 \text{Gd}_2\text{O}_3 - 3 \text{Eu}_2\text{O}_3$ (PCBGO sample) and $15\text{QS} - 57 \text{P}_2\text{O}_5 - 15 \text{CaO} - 5 \text{BaO} - 5 \text{GdF}_3 - 3 \text{Eu}_2\text{O}_3$ (PCBGEF sample). Alumina containers were provided as a place to melt the samples, and an electric furnace was used to heat the materials to form glass. The materials were weighed using digital scales according to the specified mass. The materials were mixed homogeneously with a spatula and stirred manually in an alumina crucible container. The alumina container was placed in a jar containing silica gel to reduce the water vapor in the material. The materials were left for 24 hours in a vacuum chamber. After that, the evenly mixed materials are placed in an electric furnace at a temperature of 1200°C for 3 hours until all materials melt. This melting process is called the melt quenching method [18]. After all the materials have melted and good homogeneity has been achieved, the glass material is poured into a rectangular stainless steel mold. The molded samples are annealed for 3 hours at a temperature of 500°C and slowly cooled to room temperature. Glass samples that were not cracked and had good transparency were cut into $1 \text{ cm} \times 0.3 \text{ cm} \times 1.5 \text{ cm}$ pieces and polished using a polishing

machine. The polished samples were analyzed for their physical properties and characterized using FTIR and XRD [19].

RESULTS AND DISCUSSION

Glass Sample

Samples that have been successfully prepared with a combination of Gadolinium Oxide (PCBGO) and Gadolinium Fluoride (PCBGEF) are shown in Figure 1.

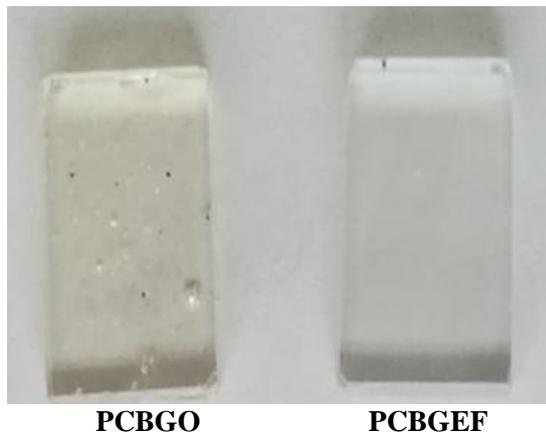


Figure 1. View of Eu:Silica-phosphate glasses.

Figure 1 shows that the two samples exhibit color differences. PCBGO is opaque white, while PCBGEF is clear white. This difference is very important to minimize unwanted light scattering and refraction during optical testing, resulting in more accurate measurements of optical properties such as refractive index, absorption, and luminescence [20]. The PCBGEF sample has the potential to be superior for optical applications such as lasers or signal amplifiers due to its better transparency, which can increase the luminescence efficiency of Eu^{3+} ions [21].

Physical Properties of Glass Medium

Table 1 below shows the physical properties of Eu:Silicate Phosphate glass medium measured for two types of glass: PCBGO and PCBGEF (each glass doped with Europium ions, Eu^{3+}).

Table 1. Physical properties of Eu:Silica-phosphate glass medium.

Physical parameter	Glass sample	
	PCBGO	PCBGEF
Molar weight (g/mol)	134.6805	127.2678
Density (gr/cm^3)	3.0982	2.8833
Molar volume (cm^3/mol)	43.4706	44.1396
Ion concentration, $N \times 10^{22}$ (ion/cm^3)	4.156	4.093
Polaron radius $\times 10^{-8}$ (Amstrong)	5.3993	5.4269
Inter nuclear distance $\times 10^{-8}$	1.34	1.3468
Field strength, $F \times 10^{17} \text{ cm}^2$	2.02	2
Refractive index (n)	1.5634	1.5409
Molar refractivity (R_m)	14.1265	13.8681
Molar electronic polarization $\times 10^{-24}$	5.6029	5.5004
Metallization criteria (M)	0.675	0.6858
Reflection loss ρ %	4.8306	4.5317
Dielectric constant (ϵ)	2.4442	2.3744
Thickness (mm)	3	3

As shown in Figure 2, the density of the glass compound decreased slightly. This decrease was caused by differences in sample composition between Gd_2O_3 and GdF_3 . The GdF_3 content was able to change the molar weight, hardness, refractive index, ion concentration, dielectric constant, molar refractivity, oxide ion capacity, and reflection loss. In the samples studied, the relative molecular mass of Gd_2O_3 was 362.497 g, while that of GdF_3 was 214.244 g, indicating that the decrease in Gd_2O_3 concentration replaced GdF_3 , thereby causing a decrease in relative density [22].

Analysis of physical parameters in PCBGO (15QS - 57 P_2O_5 - 15 CaO - 5 BaO - 5 Gd_2O_3 - 3 Eu_2O_3) and PCBGEF (15QS - 57 P_2O_5 - 15 CaO - 5 BaO - 5 GdF_3 - 3 Eu_2O_3) glass samples. The molar weight decreased from 134.6805 g/mol to 127.2678 g/mol due to the substitution of Gd_2O_3 with GdF_3 , which has a lower molar mass, causing the density to also decrease from

3.0982 g/cm³ to 2.8833 g/cm³ [23]. The molar volume increased from 43.4706 cm³/mol to 44.1396 cm³/mol due to looser fluoride bonds, affecting the increase in polaron radius from 5.3993 Å to 5.4269 Å and the inter-nuclear distance from 1.3400 to 1.3468 [24]. The ion concentration decreases slightly from 4.1560 ions/cm³ to 4.0930 ions/cm³ as the density decreases, while the field strength remains stable at 2.00 cm⁻². The refractive index decreased from 1.5634 to 1.5409, followed by a decrease in molar refractive index from 14.1265 to 13.8681 and electron polarization from 5.6029 to 5.5004, reflecting slightly reduced optical properties due to less polar fluoride bonds. The metallization criterion increased from 0.6750 to 0.6858, while the reflection loss increased from 4.8306 to 4.5317, and the dielectric constant decreased from 2.4442 to 2.3744 due to lower polar properties. The thickness remains constant at 3.0000 mm. These changes indicate that the substitution of Gd₂O₃ with GdF₃ increases the structural volume and modifies the optical and photoluminescent properties, with the potential for increased red emission efficiency for optoelectronic applications [25].

Properties of Medium Glass Structures

Glass is one of the amorphous materials widely used in various industrial applications, such as optics, electronics, and biomedicine, thanks to its unique physical properties, including transparency, hardness, and corrosion resistance [26]. However, a deep understanding of the structure of glass media at the atomic and molecular scales is key to optimizing these properties. Glass structures, which are non-crystalline or amorphous, do not have a long-range periodic arrangement like crystals, thus requiring advanced analysis techniques to reveal their characteristics [27]. In this study, Fourier Transform Infrared (FTIR) spectroscopy and X-ray diffraction (XRD) were the main tools used to investigate the structural properties of glass media.

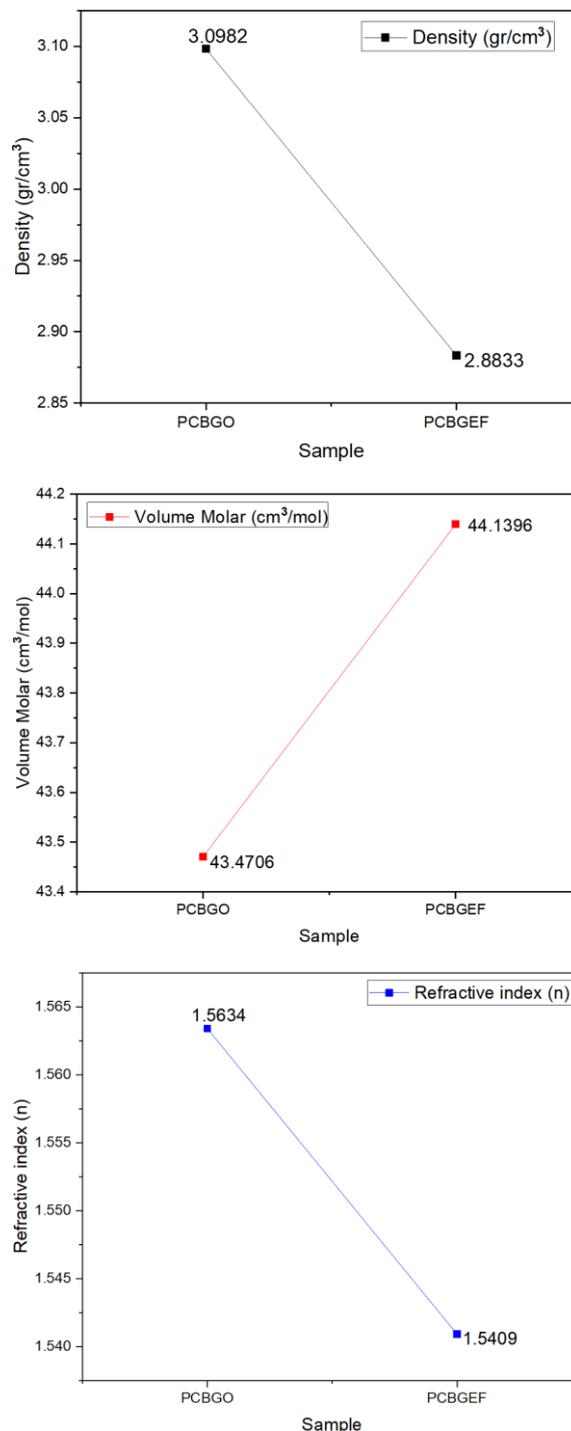


Figure 2. Density, molar volume, and refractive index graphs of Eu:Silico-phosphate glass medium.

Fourier Transform Infrared (FTIR)

FTIR spectroscopy analysis was performed on two samples, namely PCBGO and PCBGEF. Figure 3 shows the infrared transmittance (%) spectrum in the form of wave numbers at a distance of 500 – 3500 cm⁻¹.

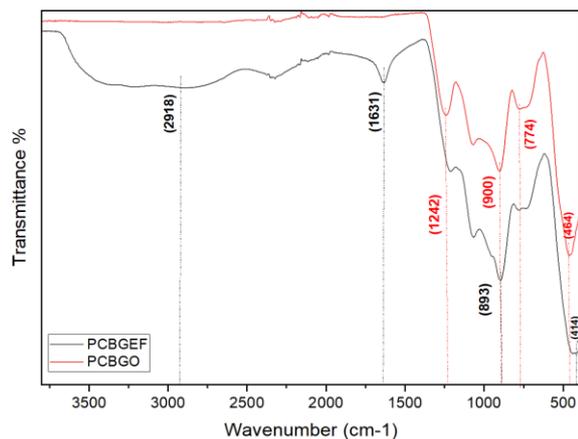


Figure 3. FTIR Spectrum of Eu:Silica-phosphate.

Table 2. FTIR spectral peak of Eu:silico-phosphate glass medium.

Wave number range	Interpretation	Ref.
2800 – 3400	Stretching vibration O–H	[28]
1600 – 1650	Extended vibration C=O	[29]
1220 – 1290	Asymmetric vibration pada PO ₃	[30]
800 – 1200	Vibration Si-O-Si or Si-O (silica matrix)	[31]
400 – 790	Vibration Si–O-Si and Si–O-Eu	[32]

At a distance of 2800 – 3000 cm⁻¹, both samples showed CH wave peaks, confirming the presence of organic groups in the silica matrix. However, the PCBGEF sample has a significant additional peak at 1600 – 1650 cm⁻¹ (peak at 1631 cm⁻¹), which can be associated with C=O or C=C stretching vibrations. In addition, the peak at a wavenumber of 1200–1500 cm⁻¹ (peak at 1242 cm⁻¹) indicates CH height, which is also only detected in PCBGEF, indicating further chemical modification [33]. The spectral differences between PCBGO and PCBGEF indicate that GdF₃ affects the chemical structure of the silica matrix, which may contribute to the properties of the glass material.

X-Ray Diffraction (XRD)

The X-ray diffraction (XRD) methodology can be used to determine crystallite dimensions

using the Scherrer equation, which is based on the observed diffraction peak width. XRD serves to evaluate the degree of crystallinity in the silica matrix, an important factor due to the influence of amorphous or crystalline properties on the matrix's capacity to facilitate energy transfer to Eu³⁺ ions [34].

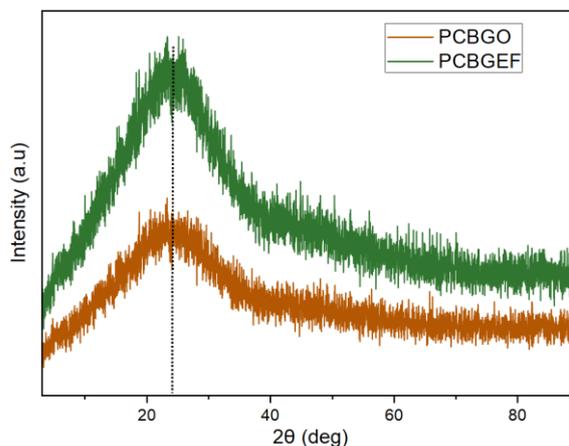


Figure 4. X-ray diffraction pattern of Eu:Silica-phosphate glass.

The X-ray diffraction (XRD) pattern of Eu:Silica-phosphate glass, as shown in Figure 4, shows broad bands indicating the presence of short-range atomic order in the glass structure [35]. Analysis of the XRD data did not reveal any sharp diffraction peaks, but only showed broad humps around an angle of 24° (2θ), reflecting the amorphous nature of the material [36]. These findings confirm that Eu³⁺ ions in Eu:SilicaPhosphate glass have amorphous characteristics.

CONCLUSION

This study successfully revealed the physical properties and structure of silico-phosphate glass medium synthesized from Huta Ginjang quartz sand, with Europium (Eu³⁺) ion doping and variations in the use of Gadolinium oxide (Gd₂O₃) and Gadolinium fluoride (GdF₃) as modifiers. XRD analysis results show that the addition of Gd₂O₃ tends to maintain a more stable amorphous structure compared to GdF₃, where samples with GdF₃ show a slight increase in diffraction peak intensity, due to the influence of fluoride ions on the glass network.

From FTIR spectroscopy, the spectrum revealed changes in the main chemical bonds, indicating modification of the silicophosphate network by high silica quartz sand from Huta Ginjang. Samples with Gd₂O₃ displayed broader bands and higher intensity, indicating an increase in the degree of polymerization and structural density, while GdF₃ produced narrower bands with a decrease, due to the fluorination effect that reduced network rigidity. This research opens up opportunities for the development of glass materials based on local Indonesian resources, with recommendations for further research on optimizing doping concentrations to improve radiation efficiency and thermal resistance.

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