Structural Properties of Gd3+/Eu3+ Double Doped in Calcium Phosphate Glass System Based on "Huta Ginjang" Quartz Sand

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Abstract. Quartz sand (Quartz Sand-QS) is an abundant natural material in Indonesia, specifically in Huta Ginjang, North Sumatra Province. This material has great potential as a basic material for the development of glass systems for medical imaging (X-ray imaging) applications. Synthesis and preparation of Huta Kidney quartz sand-based glass medium and di-double doped phosphate glasses with Gd^{3+} and Eu^{3+} ions (POCaGE) were performed for X-ray radiation scintillator applications. The composition of the glass system follows the chemical formula (15-x) QS-60P_2O_5-15CaO-5BaO-5Gd_2O_3-xEu_2O_3 (where x = 0; 0.5; 1.0; 1.5; 2.0 mol%). Samples were prepared using the melt-quenching technique at a melting temperature of 1200 ^oC and a glass system with good homogeneity and transparency was obtained. The structural properties of the glass medium were observed by using XRD and FTIR to find out the diffraction patterns and clustering of network functions in the glass structure. Based on the results of observations it is known that the quartz sand glass medium of Huta Kidney and calcium phosphate di-double doping with Gd^{3+}/Eu^{3+} ions (POCaGE) has an amorphous structure which indicates that the properties of the medium after doped are intact as glass without any crystalline properties in it. In addition, the FTIR spectrum shows a variety of absorption bands, indicating the existence of functional bonds and clusters in the glass network.

Keywords: Glasses system; quartz sand; double doping Gd^{3+}/Eu^{3+}.

1 Introduction

Phosphate-based glasses have garnered significant attention in materials science due to their versatile properties, including low melting temperatures, high chemical durability, excellent optical transparency, and biocompatibility, making them suitable for applications ranging from nuclear waste storage to biomedical implants and optoelectronic devices [1]. Among

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these, calcium phosphate glasses stand out for their ability to incorporate high concentrations of rare-earth (RE) ions, such as Gadolinium (Gd^{3+}) and Europium (Eu^{3+}) [2], which enhance their optical and luminescent properties. The incorporation of quartz sand (QS) as a primary component in glass formulations introduces a cost-effective and abundant resource, further expanding the potential for large-scale production [3]. This study introduces a novel calcium phosphate glass system based on quartz sand from Huta Ginjang, double-doped with Gd^{3+} and Eu^{3+} ions, with the chemical composition (15-x) QS-60P_2O_5-15CaO-5BaO-5Gd_2O_3-xEu_2O_3 (where x = 0; 0.5; 1.0; 1.5; 2.0 mol%). The aim is to investigate the structural and luminescent properties of this glass system, leveraged the synergistic effects of Gd^{3+} and Eu^{3+} co-doping to developed advanced materials for optoelectronic and radiation detection applications [4]. Phosphate glasses are characterized by a network of phosphate (P_2O_5), where P_2O_5 acts as the primary network former, creating a robust yet flexible structure. In the proposed composition, P_2O_5 serves as the backbone of the glass matrix, providing structural stability and enabling the incorporation of modifier ions [5]. Its high content ensures a strong covalent network, which is critical for maintaining the amorphous nature of the glass while accommodated high concentrations of Eu^{3+} ions [6-7].

Calcium Oxide functions as a network modifier, CaO also improves resistance to alkali solutions, making the glass suitable for harsh environmental conditions [8]. Barium oxide further modifies the glass network, BaO larger ionic radius compared to CaO contributes to a denser and more compact glass structure, enhancing mechanical properties and optical clarity. [9] The unique properties of this glass system could lead to innovative applications in laser technology and radiation detection, harnessing the increased luminescence of the co-doping of Gd^{3+} and Eu^{3+} ions [10]. Quartz sand primarily composed of silica (SiO_2), is a novel addition to this glass system, served as a secondary network former alongside P_2O_5 [3]. The used of quartz sand from Huta Ginjang, a local resource, introduces an economically viable alternative to high-purity silica, reducing production costs while maintaining structural integrity [3, 11-12]. The incorporation of QS also enhances the glass's thermal stability and optical transparency, critical for optoelectronic applications [13-14]. The variable QS content (adjusted by the Eu_2O_3 concentration) allows for fine-tuning of the glass's structural and optical properties, providing [15] flexibility in tailoring the material for specific applications. The double doped of Gd^{3+} and Eu^{3+} ions, introduced via Gd_2O_3 and Eu_2O_3 (0,0; 0,5; 1.0;1.5; 2.0 mol%), respectively, is a key feature of this glass system. [16]. Gadolinium ions (Gd^{3+}) act as both a network modifier and a luminescent center, enhancing the glass's structural density and dielectric properties. Gd^{3+} ions are known for their ultraviolet (UV) emission at approximately 312 nm, attributed to the $^6P_{7/2} \rightarrow {}^8S_{7/2}$ transition, make them ideal for phototherapy lamps and UV-emitting devices.

Additionally, Gd^{3+} facilitates energy transfer to co-doped ions, such as Eu^{3+}, amplified their luminescence efficiency. [17-19]. Europium ions are widely recognized for their intense red emission, particularly the $^5D_0 \rightarrow {}^7F_2$ transition at ~615 nm, which is hypersensitive to the local environment and ideal for applications in solid-state lighted and display devices. The co-doping strategy leverages the energy transfer from Gd^{3+} to Eu^{3+}, enhanced the overall luminescence intensity and enabled tunable emission properties by varyed the Eu^{3+} concentration [20-21]. The novelty of this research lies in the integration of quartz sand from Huta Ginjang into a calcium phosphate glass system, combined with the strategic double doped of Gd^{3+} and Eu^{3+} ions [22-23]. While phosphate glasses doped with Eu^{3+} ions have been

extensively studied, the used of locally sourced quartz sand as a major component is unprecedented, offered a sustainable and cost-effective approach to glass synthesis [24-25].

The specific composition (15-x) QS-60P_2O_5-15CaO-5BaO-5Gd_2O_3-xEu_2O_3 (where x = 0; 0.5; 1.0; 1.5; 2.0 mol%) allows for systematic investigation of the interplay between QS content and Eu^{3+} concentration, provided insights into the structural and luminescent behavior of the glass. The double-doped strategy, coupled with the unique properties of QS, positions this glass system as a promised candidate for advanced optoelectronic applications, including white light-emitted diodes (WLEDs), X-ray scintillators, and radiation shielded materials [26-28]. Recent research on Eu^{3+} doped phosphate glasses provides a foundation for this study. For instance, investigations into Gd^{3+} doped phosphate glasses have demonstrated increased density and UV emission intensity with higher Gd_2O_3 content, alongside improved structural rigidity. Similarly, Eu^{3+}-doped borate and boro-phosphate glasses have shown enhanced red emission with increased Eu^{3+} concentration, attributed to covalent Eu-O bonding [29-30]. [31] Studies on co-doped systems, such as Tb^{3+}/Eu^{3+} in silicate sol-gel glass-ceramics, have reported energy transfer mechanisms that enable tunable luminescence, supporting the potential of Gd^{3+}/Eu^{3+} co-doping. Additionally, research on calcium phosphate glasses doped with Dy^{3+} has highlighted their potential for X-ray imaging and temperature sensing, with efficient energy transfer from Gd^{3+} to Dy^{3+}. However, these studies primarily focus on conventional glass formers like B_2O_3 or SiO_2, without exploring the use of natural quartz sand or the specific Gd^{3+}/Eu^{3+} co-doping in a QS-based calcium phosphate matrix. [32-33]. The structural properties of phosphate glasses are influenced by the incorporation of Eu^{3+} ions, which act as network modifiers, induced depolymerization of the glass network. Fourier Transform Infrared (FTIR) spectroscopy studies have shown that Gd^{3+} ions increase the formation of NBOs, while Eu^{3+} ions enhance the covalency of the glass matrix, affected optical properties like refractive index and bandgap [34-35].

Furthermore, the luminescent properties of Gd^{3+}/Eu^{3+} co-doped glasses are anticipated to exhibit enhanced emission due to energy transfer, similar to that observed in Dy^{3+}-doped gadolinium calcium phosphate glasses. These findings underscore the potential of the proposed glass system to achieve superior optical and structural performance. This study addresses critical gaps in the literature by explored the synergistic effects of Gd^{3+}/Eu^{3+} co-doping in a quartz sand-based calcium phosphate glass. The use of Huta Ginjang quartz sand not only reduces reliance on high-purity silica but also aligns with sustainable material Potential applications include high-efficiency WLEDs, where tunable white light emission is achieved through Gd^{3+}/Eu^{3+} energy transfer, and X-ray scintillators, where the high density and luminescence of Gd^{3+}-doped glasses enhance detection efficiency. Additionally, the glass's biocompatibility and chemical durability make it a candidate for biomedical applications, such as drug delivery systems or imaging agents. The study highlights the innovative use of locally sourced quartz sand, which not only provides economic benefits but also improves the optical and structural properties of the resulting calcium phosphate glass system.

2 Method

Glass with the chemical composition (15-x) QS-60P_2O_5-15CaO-5BaO-5Gd_2O_3-xEu_2O_3 (where x = 0; 0.5; 1.0; 1.5; 2.0 mol%). was produced using high-purity chemicals (99.9%). However, quartz sand (QS) is known to contain 99% SiO_2. The materials were weighed according to the glass composition calculations, then mixed homogeneously in an alumina container, and stored in a jar contained silica gel to reduce moisture in the materials. The homogenized mixture of materials is placed in a furnace and melted at a temperature of 1200 °C for approximately 3 hours until a homogeneous molten glass is formed. After that, the molten glass is poured into a stainless steel mold, and the sample is annealed at a temperature of 500 °C for 3 hours. The resulted glass is then cut into pieces measured 1 cm x 0.3 cm x 1.5 cm.

3 Result and Discussion

3.1 Glass Pattern

The physical form of the glass medium is considered to play a very important role, so the shape of the glass is regarded as a crucial factor in the fabrication process. The glass used is selected based on criteria of very high transparency, good material homogeneity, and freedom from cracks or surface defects. Cutt and polish are carried out to ensure optimal quality at the fabrication stage. The fabrication process of the glass, both before and after the cutting and polishing stages, can be observed in Figure 1, where the condition of the glass medium after cut and polishing show in Figure 2.

Figure 1. POCaGE glass samples before cut and polished

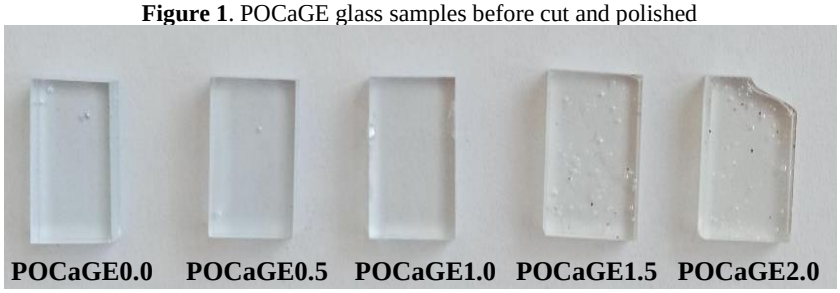


Figure 2. POCaGE glass samples after cut and polished

3.2 Physical Properties

The physical attributes that characterize the POCaGE glass medium comprise a diverse array of critical parameters, which include, but are not restricted to radius, the spatial distance between the cores, electric field strength, dielectric permittivity, molar refractivity, ionic polarizability criteria for metallization, and the measurable outcomes pertaining to reflection loss. All of these elements are meticulously and thoroughly elucidated within the scope of Table 1.

Table 1. Physical properties calculation of glass medium (15-x) QS-60P_2O_5-15CaO-5BaO-5Gd_2O_3-xEu_2O_3 (with x= 0; 0.5; 1.0; 1.5 and 2.0 mol%)

Measurement parameters	Glass Samples				
	POCaGE 0.0	POCaGE 0.5	POCaGE 1.0	POCaGE 1.5	POCaGE 2.0
Weight molar, *M* (g)	128.381	129.840	131.299	132.759	134.218
Density (g/cm^3)	2.838	2.864	2.909	2.921	2.951
Molar volume (cm^3/mol)	45.231	45.330	45.129	45.451	45.481
Ion concentration, N x 10^{20} (ion/cm^3)	-	0.664	1.330	1.990	2.650
Polaron radius x10^{-8} (Å)	-	9.950	7.880	6.900	6.270
Inter nuclear distance x 10^{-7}	-	2.470	1.960	1.710	1.560
Field strength, F x 10^{16} cm^2	-	0.596	0.949	1.240	1.500
Refractive index (n)	1.539	1.541	1.543	1.546	1.570
Molar refractivity (R_m)	14.182	14.244	14.229	14.396	14.930
Molar electronic Polarization x 10^{-24}	5.630	5.650	5.640	5.710	5.920
Metallization Criteria (M)	0.686	0.685	0.685	0.683	0.672
Reflection loss (R) %	4.515	4.533	4.562	4.602	4.924
Dielectric constant (Ɛ)	2.370	2.375	2.382	2.391	2.466

Table 1 shows that the physical properties of the glass medium are influenced by the addition of Europium ions. Density is considered one of the important parameters in determining glass quality, where density measurements were conducted using the Archimedes method with the glass medium submerged in water [36]. Density, refractive index, ion concentration, reflection loss and dielectric constant have shown an increasing trend with the increase in Eu^{3+} ion concentration [37]. The increase in molar weight is due to the decrease in Quartz Sand (QS) as the amount of Eu_2O_3 increases, since Eu_2O_3 has a higher molar mass than QS [38]. The polaron radius and inter nuclear distance decrease as the concentration of RE ions increases, indicating a reduction in free space and a more compact glass structure. The material exhibited insulating behavior with metallization; however, in this study, the average value is approximately 0.685. Generally, values closer to zero suggest conductivity, while those nearer to one indicate insulation**.**

3.3 Structural Propterties

X-ray diffraction (XRD) is widely used as an analytical technique for the characterization of sample structures. The POCaGE glass spectrum displayed in Figure 3 is shown within the range of 10^o to 90^o, where the spectrum shape is observed to align with previous research conducted by [39], and no sharp peaks are found throughout the observed 2θ diffraction angle region. The XRD data, which exhibits non-sharp diffraction peaks but broad humps observed at around the 2θ angle, has been utilized to indicate the characteristics of an amorphous material [40]. A wide scattering at low angles is noted, which is a typical feature of structural disorder. This suggests that all prepared samples possess an amorphous structure, a

characteristic of glass materials. Thus, it can be concluded that all prepared samples are glass materials with an amorphous structure. This condition is believed to be caused by the glass composition being perfectly melted in the furnace and the homogeneous mixing of the chemical composition.

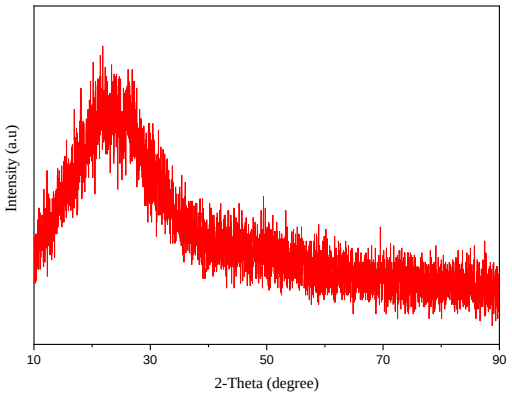


Figure 3. XRD spectra of POCaGE1.5 glasess

3.4 Optical Propterties

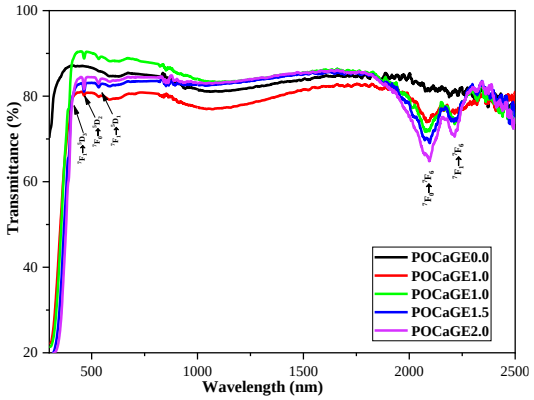


Figure 4. Transmitansi Spectra of Eu^{3+} doped QS-P_2O_5-CaO-BaO-Gd_2O_3-Eu_2O_3

The Transmitansi Spectra of Eu^{3+} doped QS-P_2O_5-CaO-BaO-Gd_2O_3-Eu_2O_3 glass are shown in the Figure 4 transmittance indicates how much light passes through a material higher values signify greater transparency [41]. All samples exhibit a sharp increase in transmittance below 400 nm, peaking and stabilizing in the 500–2000 nm range. [42] Variations in POCaGE composition affect transparency levels, with maximum transmittance reaching 70–90% in the visible to near-infrared range [43]. A slight decrease in transmittance occurs beyond 2000 nm due to characteristic absorption of the glass or impurities in the infrared band. The cut-off wavelength, marking the transition from opaque to transparent behavior, occurs around 350–400 nm. Below this range, transmittance is very low due to significant light absorption. Beyond the cut-off wavelength, the glass becomes sufficiently transparent for optical applications, such as optical media or transparent filters [44].

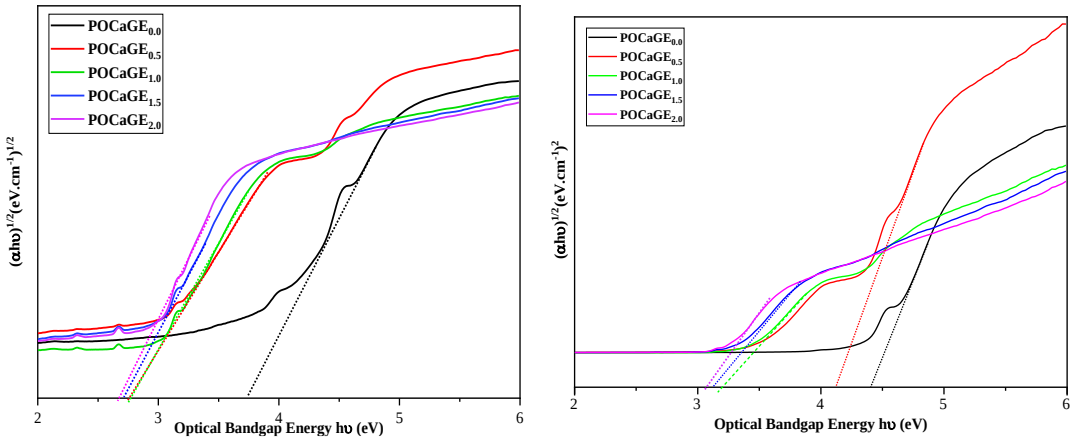
The absorption coefficient α(ν) was calculated from the absorbance (A) using the following equation:

$$\alpha(\nu) = \left(\frac{1}{d}\right) ln\left(\frac{I_0}{I}\right) = 2.303\left(\frac{A}{d}\right)$$

where A is the absorbance at frequency n and d is the thickness of the sample. For an absorption by an indirect transition, the equation takes the form:

$$E_{opt} = h\nu - \left(\frac{h\nu}{B}\right)^{1/2}$$

where B is a constant called band tailing parameter, hν is the incident photon energy and E_{opt} is the optical band gap energy. Using the above equations, by plotting $(ahv)^{1/2}$ as a function of photon energy hν, one can find the optical band gap energy (E_{opt}) for indirect transitions and direct by extrapolating the linear region of the curve to the hν axis [45] and are shown in Figure 5.



(a) (b)

Figure 5. (a) Indirect and (b) Direct optical band gap of POCaGE glass samples

Table 2. Direct and indirect optical band gaps of the various glass samples

Glass samples	Indirect (eV)	Direct (eV)
POCaGE0.0	3.75	4.46
POCaGE0.5	2.78	4.17
POCaGE1.0	2.77	3.28
POCaGE1.5	2.72	3.21
POCaGE2.0	2.67	3.15

4 Conclusion

Europium-doped phosphate glasses were successfully synthesized via the melt-quenching technique and characterized using various spectroscopic methods, including X-ray diffraction (XRD) and Fourier Transform Infrared (FTIR) spectroscopy. The results confirm that the prepared samples exhibit complete amorphous nature, as evidenced by the absence of crystalline peaks in XRD patterns. Physical properties such as density, refractive index, reflection loss, and dielectric constant demonstrated a consistent increasing trend with rising Eu^{3+} ion concentration, reflecting enhanced ion packing and network modifications. Structurally, Eu^{3+} ions act as network modifiers in the phosphate glass matrix, promoting depolymerization and the formation of non-bridging oxygens (NBOs), as revealed by FTIR analysis. These findings highlight the potential of Eu^{3+}-doped phosphate glasses for applications in optical and luminescent devices, warranting further exploration of their luminescent efficiency and stability.

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