

THE STUDY AND INVESTIGATION OF DYSPROSIUM-DOPED GLASSES MADE FROM SUGARCANE LEAVES FOR WHITE LIGHT EMITTING MATERIAL

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Abstract

The physical, optical, and photoluminescence properties of Dy₂O₃-doped glasses, synthesized from sugarcane leaves using a simple melt-quenching technique, were systematically investigated in this study. Various characterization techniques such as XRD, density, refractive index, absorption spectra, FTIR, and PL spectra were employed to evaluate their properties. The absorption spectra of Dy³⁺-doped glasses exhibited prominent peaks at wavelengths of 451, 798, 895, 1085, 1261, and 1673 nm. Photoluminescence analysis exhibited emission and excitation spectra, with the highest peak observed at a concentration of 1.5 mol%. Additionally, the values of decay time, the CIE 1931 chromaticity, CCT, and the Y/B ratio were assessed. The findings underscore the potential of the SCDy glasses for the advancing of white-light applications

Keywords: Sugarcane Leaves; Dysprosium; Glass; Photoluminescence; White-Light Emitting Materials

Introduction

Sugarcane holds significant economic importance in Thailand as it serves as the key source for both sugar production and renewable energy (Paopun *et al.*, 2022). Consequently, it plays an important role in Thai's agricultural crop system. The method of harvesting sugarcane results in a large amount of sugarcane leaves remaining in the field that consisting of both fresh and dried leaves with weights of 0.975 and 1.531 kg/m² (Lontom *et al.*, 2009), respectively. Thai farmers always burn their

fields immediately after harvesting, as it is the quickest and simplest method to eliminate the leaves remaining in fields. However, it contributes to environmental issues such as air pollution, including small dust particles, smoke, ash, soot, and PM 2.5 emissions (Lontom *et al.*, 2009; Paopun *et al.*, 2022).

Glass is an inorganic object that has an amorphous structure, and it is transparent and luster (Ichoja *et al.*, 2020). It comes with various types

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depending on its main components, including silicates, borates, phosphates and others. It finds applications in the wide range of areas such as containers, windows, fiber optics, scientific materials, engineering, applications in the arts, etc. (Kaewkhao *et al.*, 2015; Chimalawong *et al.*, 2019; Kaewjaeng *et al.*, 2019; Pintori *et al.*, 2022). Glass is an inorganic object that has an amorphous structure, and it is transparent

Lanthanides (Ln) element performed as excellent activators to emit sharp fluorescence in bands from the UV to the infrared region, and Ln have a 4f-4f or 5d-4f emission shift. Dy^{3+} is assumed as a created for white light generation because its emission in the UV-visible regions (Himamaheswara Rao *et al.*, 2018, Pawade *et al.*, 2020, Söldner *et al.*, 2020). Dy^{3+} is an excellent optical activator and it gives simultaneous blue, yellow and red emissions. It can also be useful for laser applications and optical amplification (Nagaraja Naick *et al.*, 2019). White light emitting diodes (w-LEDs) have become most popular in recent years due to their exceptional qualities, which including high brightness, low power consumption, extended lifespan, increased efficiency, environmental friendliness, outstanding dependability, and low-temperature efficiency (George *et al.*, 2019; Ahmad *et al.*, 2020; Monisha *et al.*, 2020). According to the literature examining the compositions of sugar cane leaves using XRF technique, silica and alumina were identified as the predominant components (Yotkuna *et al.*, 2021). Therefore, sugar cane leaves could potentially serve as an alternative to silica, chemical powder utilized in glass melting process for both soda lime silicate glass and borosilicate glass.

In this research, the sugarcane leaves were used as silica source to produce the dysprosium-doped borosilicate glasses. Furthermore, the research investigated the physical, optical and

luminescence properties of these glasses, with the aim of determining their potential as the white-light emitting materials.

Experimental Procedure

Sugarcane leaves collection and characterization

Dried sugarcane leaves were collected from a field in Nakhon Pathom province and were then divided into five groups, each weighting 1 kg. These groups were then subjected to calcination at different temperatures in an electric furnace. The temperatures used for calcination were: Raw, 400°C, 600°C, 800°C and 1,000°C, respectively. The chemical compositions of the calcined samples were investigated using an X-ray fluorescence spectrometer (PANalytical, Minipal-4) and the results were tabulated in Table 1. After calcination each group yielded approximately 50 grams of ash. From Table 1, it is evident that the main components of sugarcane leaves were SiO_2 and CaO, respectively. Moreover, the SiO_2 content increased with increasing calcination temperature. Hence, the sugarcane leaves calcined at 1000°C was selected to produce the dysprosium-doped borosilicate glasses.

Glass production method

In this procedure, the sugarcane leaves ash which, was calcined at 1000°C, was utilized as SiO_2 to prepared dysprosium-doped borosilicate glasses with nominal composition 55 SiO_2 : 25 B_2O_3 : 10 Na_2O : 10CaO: x Dy_2O_3 (where x denotes the concentration Dy_2O_3 , ranging from 0.0, 0.5, 1.0, 1.5, 2.0 and 2.5 mol%), were produced. The details for batch preparation and glass preparation methods are provided in our previous work (Mahingsa *et al.*, 2023). The as-prepared glasses (referred as the SCDy series) are transparent and exhibit slightly yellow color, as depicted in Figure 1.

Table 1. The chemical compositions (in wt%) of sugarcane leaves at different calcination temperatures

Compounds	Calcination temperatures (°C)				
	Raw	400°C	600°C	800°C	1000°C
SiO_2	69.60	69.74	69.82	69.89	69.93
CaO	17.06	17.15	16.42	16.38	18.57
K_2O	4.43	4.38	4.40	3.20	1.72
SO_3	2.64	2.52	3.13	3.54	2.44
P_2O_5	2.15	2.08	2.03	2.20	2.31
MgO	2.00	2.13	2.28	2.30	2.36
Al_2O_3	1.40	1.35	1.33	1.70	1.70
Fe_2O_3	0.54	0.48	0.44	0.54	0.73
MnO	0.08	0.08	0.08	0.09	0.10
TiO_2	0.07	0.06	0.06	0.11	0.09
CuO	0.02	0.02	0.02	0.03	0.04
ZnO	0.01	0.01	0.01	0.02	0.02
Total	100.00	100.00	100.00	100.00	100.00

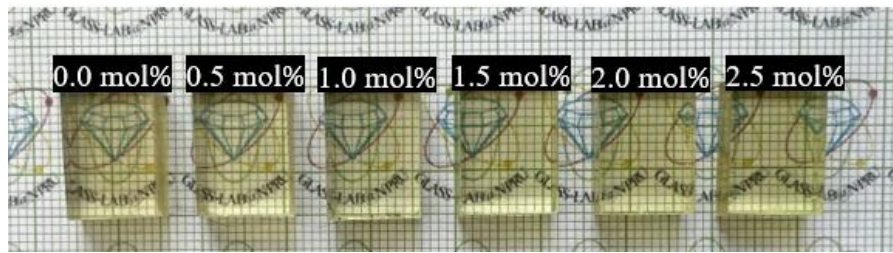


Figure 1. The SCDy glasses doped with different concentrations of Dy_2O_3

Table 2. The density, molar volume, and refractive index of the SCDy glasses

Parameters	0.0SCDy	0.5SCDy	1.0SCDy	1.5SCDy	2.0SCDy	2.5SCDy
Density [g/cm^3]	2.8455	3.0173	3.0181	2.9286	3.0342	3.0293
Molar volume [cm^3/mol]	21.8798	20.7141	20.8836	21.6880	21.4175	21.4461
Refractive index [-]	1.5638	1.58206	1.58082	1.57444	1.58082	1.5832

Samples characterization

The examination of density, molar volume, and refractive index were conducted at room temperature using the relationships documented in the literature (Zhao *et al.*, 2016), with the corresponding results presented in Table 2. The FTIR analysis was employed the structural groups presented in the glass network, which could be categorized into distinct ranges (Torres-Carrasco *et al.*, 2016; Zhao *et al.*, 2016). Absorption spectra analysis of the SCDy glasses was performed using a UV-Vis-NIR spectrometer (Shimadzu, UV-3600). The photoluminescence characteristics were studied at room temperature using xenon lamp for UV excitation. The decay time was investigated by a fluorescence spectrofluorometer (Hitachi, F-4600). Additionally, the CIE 1931 chromaticity was used to define the luminescent color achieved from the SCDy glasses.

Results and Discussion

Physical properties

From Table 2, the density tended to increase with adding Dy^{3+} because molecular mass of Dy_2O_3 is heavier than SiO_2 . The molar volume has a rising trend with increasing concentration of Dy_2O_3 . This demonstrates obviously that the number of non-bridging oxygens (NBOs) increased with dopant concentration and leading to an extension of glass network (Chimalawong *et al.*, 2012; Zhao *et al.*, 2016). The refractive index ranged from 1.5638 - 1.5832 and tended to increase, consistent with the density trend. This relationship is attributed to the fact that refractive index depends on the density and degree of polarization of an atom in glass sample (Mahingsa *et al.*, 2023).

The structural group has been examined using FTIR analysis and peaks appeared in the spectrum (in Figure 2) corresponding to symmetric stretching vibrations of Si-O bonds (at 687 cm^{-1}), stretching vibrations of Si-OH bonds (at 954 cm^{-1}), and antisymmetric stretching vibrations of Si-O-Si bonds (at 1360 cm^{-1}) confirm the presence of SiO_2 as the main component of the glass samples. Additionally, the presence of Si-OH bonds contributes to the hydrophilicity of the SiO_2 aerogel (Shi *et al.*, 2022).

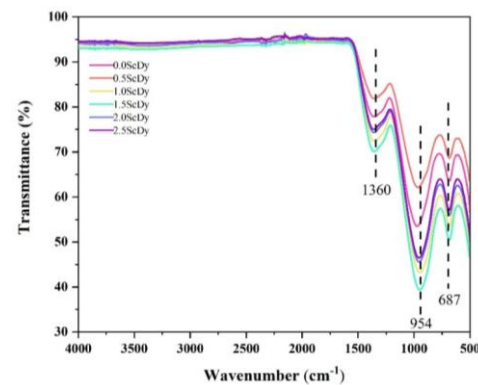


Figure 2. The SCDy glasses doped with different concentrations of Dy_2O_3

Optical properties

In Figure 3, the absorption spectra of the SCDy glasses were investigated over wavelength of 200 - 2500 nm and performed in Figure 3. Notably, absorption bands attributed to Dy^{3+} ions consistently appear at almost identical wavelengths across all samples, with slight variations in intensity. The absorption spectra consist of six

broadened bands at around 451, 798, 895, 1085, 1261, 1673 nm, corresponding to transition of from ground state ${}^6\text{H}_{15/2}$ to various excited states ${}^4\text{I}_{15/2}$, ${}^6\text{F}_{15/2}$, ${}^6\text{F}_{7/2}$, ${}^6\text{F}_{9/2}$, ${}^6\text{F}_{11/2}$ and ${}^6\text{H}_{11/2}$, respectively. It is observed that an intense absorption band located at 1261 nm of the electromagnetic spectrum due to the electronic transition of ${}^6\text{H}_{15/2}$ to ${}^6\text{F}_{11/2}$.

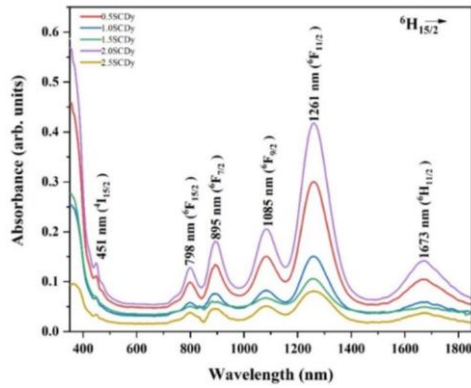


Figure 3. The absorption spectra of the SCDy glasses doped with Dy_2O_3

The photoluminescence excitation and emission spectra of the SCDy glasses were presented in Figure 4(a) and 4(b), accordingly. The excitation spectra were recorded in the range of 300 - 500 nm by monitoring the emission at 578 nm. The six excitation bands centered at 351, 365, 390, 426, 453, 471 nm. Among the mentioned excitation wavelengths, the excitation band at 390 nm exhibits the maximum luminescence intensity as compared to the others band. Hence, the wavelength at 390 nm was used to record the emission and decay time measurements. In Figure 4(b), three characteristic emission peaks at 485 (blue), 578 (yellow) and 666 nm (red) can be noticed, corresponded to the transition from ${}^6\text{F}_{9/2}$ to excited states ${}^6\text{H}_{15/2}$, ${}^6\text{H}_{13/2}$ and ${}^6\text{H}_{11/2}$, respectively. The yellow emission at 578 nm exhibits a stronger intensity compared to blue emission located at 485 nm. While the emission in red region demonstrates a negligible intensity when compared to the other two observed emission. The emission intensity increased with rising of Dy^{3+} concentration up to 1.5 mol%, after that emission intensity decreased due to the occurrence of concentration quenching effect (Torres-Carrasco *et al.*, 2014).

The color coordinates (x, y) within the CIE 1931 chromaticity, were employed to determine the characteristic emission color of the glasses, using the emission spectra profiles. The obtained values of the CIE 1931 coordinates were summarized in Table 3 for the SCDy glasses. The locations of these

coordinates well fall within white region of the CIE 1931 diagram (as seen in Figure 5). This observation indicates that all glasses emit white light upon excitation with 390 nm, rendering them suitable for the production of white light emitting materials.

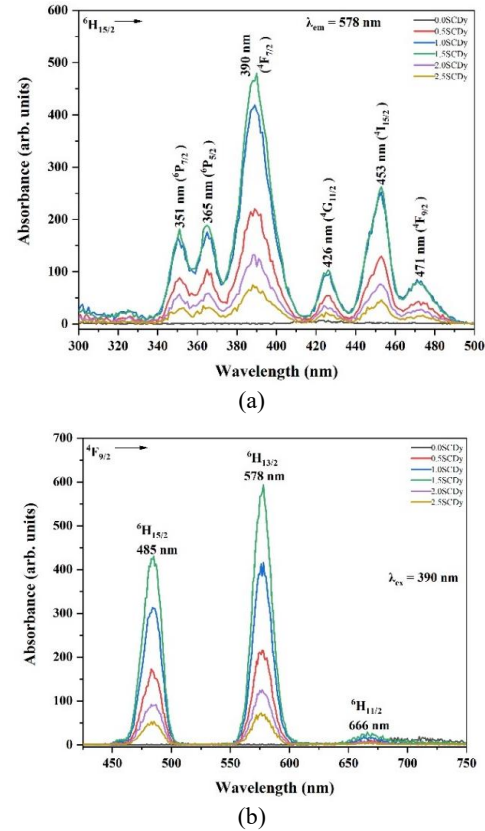


Figure 4. The excitation (a) and emission (b) spectra of the SCDy glasses doped with Dy_2O_3

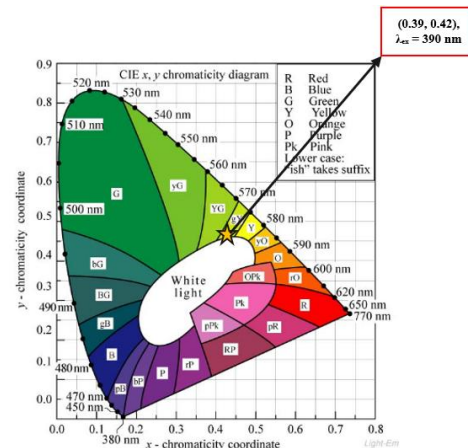


Figure 5. The typical CIE 1931 chromaticity of the 1.5SCDy glass sample

Table 3. The values of color coordinates in the CIE 1931 chromaticity diagram, CCT, and Y/B intensity ratios for the SCDy glasses

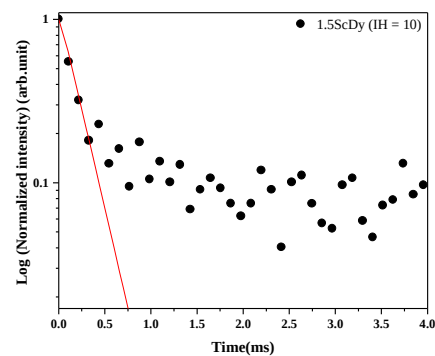
Glass samples	CIE 1931 chromaticity values		CCT (K)	Y/B intensity ratio
	x	y		
0.5SCDy	0.3751	0.4050	3318	1.378
1.0SCDy	0.3742	0.4028	3342	1.327
1.5SCDy	0.3768	0.4051	3232	1.250
2.0SCDy	0.3739	0.4044	3373	1.385
2.5SCDy	0.3759	0.4071	3298	1.368

Table 4. The decay time values of the SCDy glasses doped with different concentration of Dy₂O₃

Parameter	0.0SCDy	0.5SCDy	1.0SCDy	1.5SCDy	2.0SCDy	2.5SCDy
Decay time, τ [ms]	-	0.311	0.223	0.175	0.166	0.100

The correlated color temperature (CCT) is a parameter used to describe the perceived color of light emitted from a source in comparison to that emitted from a reference source at a given temperature and expressed in kelvin (K). The CCT can be computed by McCamy's equation (Talewar et al., 2019). In addition, the yellow to blue intensity ratio (Y/B) is another parameter to indicate the capability to generate blue or white light (Hernández-Andrés *et al.*, 1999), influenced by the Dy³⁺ concentration in the studied materials. The calculated values of CCT and Y/B ratios were summarized in Table 3. The calculated CCT values for all SCDy glasses were found to be in the range of 3298 - 3373 K, which is regarded as a warm white light in appearance.

The decay profiles of the SCDy glasses were measured using excitation at 390 nm and emission at 578 nm as reported in Table 4. These decay curves display a consistency decline with rising Dy₂O₃ concentration. As the Dy₂O₃ concentration increases, the decay time decrease, suggesting energy transfer via cross-relaxation between Dy³⁺ ions. Consequently, higher Dy³⁺ concentrations in the SCDy glasses lead to shorter decay time was observed (Kaewnuam *et al.*, 2017). As illustrated in Figure 6, the IH model fitting for S = 10 exhibited excellent agreement with the experimental data, confirming the existence of quadrupole-quadrupole interactions between Dy³⁺ ions (Shoaib *et al.*, 2020). A decrease in decay time was observed with increasing Dy³⁺ concentration, while the energy transfer parameter (Q) showed a corresponding increase. This rise in Q values is associated with the higher Dy³⁺ ion concentration. The observed reduction in radiative decay time is attributed to cross-relaxation and resonance energy transfer processes occurring between Dy³⁺-Dy³⁺ ions within the glass matrix (Samuhara *et al.*, 2023, Vijayakumar *et al.*, 2014).

**Figure 6. The typical decay time of the 1.5SCDy glass sample**

Conclusions

In this study, dysprosium-doped borosilicate glasses were prepared using calcined sugarcane leaves at 1000°C as silica source, aiming to evaluate their potential in white light emitting materials. The density, molar volume and refractive index display the increasing trend with increasing Dy³⁺ ion concentration. The absorption spectra revealed six characteristic absorption bands corresponding to the transition from ⁶H_{15/2} to ⁴I_{15/2}, ⁶F_{15/2}, ⁶F_{7/2}, ⁶F_{9/2}, ⁶F_{11/2} and ⁶H_{11/2}. The photoluminescence of the SCDy glasses performed the highest spectra under 390 nm excitation and 578 nm emission at a concentration of 1.5 mol% of Dy₂O₃. The decay time, derived from luminescence spectra, decreased with increasing Dy³⁺ concentration. Furthermore, the CIE 1931 chromaticity, CCT values, and Y/B ratio confirmed the emitted light fall within white region, indicating that the SCDy glasses could be suitable candidate for white light emitting devices.

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