

Enhanced IR and UV Shielding Performance of Leucoxene-CuS Composite-Based Paint Coatings

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Abstract

The increasing energy consumption of heating and cooling systems underscores the need for advanced materials that improve the thermal insulation of building envelopes, particularly windows. This study develops and characterizes a novel paint coating for glass that integrates UV-reflective and IR-responsive optical properties. The coating is based on a composite of leucoxene, a natural TiO₂-rich mineral that acts as a UV-reflective component, and copper sulfide (CuS) nanoparticles, synthesized via co-precipitation, which serve as potent IR absorbent enhancers. Composites with varying CuS-to-leucoxene weight ratios (1:9 to 5:5) were systematically analyzed using X-ray diffraction (XRD) and scanning electron microscopy (SEM). The composites were incorporated into an acrylic paint matrix and applied onto glass substrates. Optical characterization using UV-Vis-NIR spectroscopy indicated that increasing CuS content led to reduced diffuse reflectance, while higher leucoxene content contributed to improved UV reflectance. Thermal gradient behavior, evaluated under controlled IR irradiation, revealed that the 5:5 CuS-to-leucoxene composite exhibited the largest measured surface-to-interior temperature gradient, achieving a temperature difference (ΔT) of approximately 47 °C between the coated surface and the interior. Notably, the 1:9 composition demonstrated comparable performance with a lower filler content, indicating a more cost-effective alternative. These results demonstrate the potential of leucoxene-CuS composites as tunable and functional coatings for energy-efficient window and building applications.

Keywords: Nanocomposite, Leucoxene, Copper Sulfide, Radiation Protection, Optical Materials, Co-precipitation, Thermal Insulation

1. Introduction

Rapid global population growth and industrialization have escalated energy demands and environmental pollution, intensifying concerns over fossil fuel depletion. In this context, the building sector is a major energy consumer, with heating and cooling systems accounting for more than half of its total energy use [1]. Windows, due to their inherently poor thermal insulation, represent a significant source of energy loss. Solar energy comprises a substantial near-infrared (NIR) component (approximately 52%), which transmits heat through glazing. Consequently, developing transparent coatings with effective NIR shielding is critical for improving the energy efficiency of buildings and vehicles [1].

Copper sulfide (CuS), a p-type semiconductor, has emerged as a promising material for such applications due to its strong absorption in the UV and NIR regions, tunable optical properties, and stability under UV exposure [2]. For instance, Kwon et al. demonstrated that electrospun CuS/polyvinylpyrrolidone (PVP) nanofibers deposited on glass substrates exhibit strong UV-NIR absorption and effective thermal shielding while maintaining high visible light transmittance [3]. Subsequent work showed that gallium doping could further enhance the NIR shielding performance of CuS nanocrystals via localized surface plasmon resonance (LSPR) [4]. However, the particulate nature

of CuS renders it opaque, limiting visible light transmission and its application in fully transparent coatings.

Leucoxene, a naturally occurring titanium dioxide (TiO_2)-rich mineral (60–90%), offers high thermal stability, durability, and strong UV reflectance, making it a suitable component for functional coatings [4]–[7]. By combining the complementary properties of CuS (a strong NIR absorber) and leucoxene (a strong UV reflector) within a single paint matrix, it is possible to create a composite coating that provides enhanced shielding across a broader solar spectrum while maintaining desirable film properties [6]. Despite the individual merits of CuS and TiO_2 -based materials, a knowledge gap remains in the design of composite coatings that simultaneously tunable shield both UV and NIR radiation using low-cost, naturally derived mineral components. We hypothesize that combining CuS nanoparticles (NIR absorber) with leucoxene (UV reflector) in controlled weight ratios enables tunable spectral selectivity and enhanced thermal insulation performance in a practical paint coating system. Systematically varying the CuS-to-leucoxene ratio allows exploration of the trade-off between NIR absorption efficiency and UV reflectance, providing a rational pathway for optimizing the composite coating for specific energy-efficiency requirements in window and building envelope applications.

In this work, we synthesized CuS nanoparticles via a co-precipitation method and prepared leucoxene-CuS composite coatings using a commercial acrylic binder. We systematically investigated the effects of different CuS-to-leucoxene ratios on the structural, optical, and thermal insulation properties of the coatings. The influence of binder composition and film uniformity on performance was also evaluated. This study aims to optimize a functional coating system that effectively blocks UV and IR radiation for energy-efficient window applications.

2. Experimental

2.1 Materials

Copper (II) nitrate trihydrate ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$) and thiourea (NH_2CSNH_2) were purchased from Sigma-Aldrich. Ammonia solution (NH_3 , 28%) was obtained from Panreac. Ethanol ($\text{CH}_3\text{CH}_2\text{OH}$, absolute) was acquired from Merck. Diethanolamine and isopropyl alcohol were purchased from LOBA CHEMIE PVT. LTD. All chemicals were of analytical grade and used without further purification. The leucoxene mineral powder was sourced from a local supplier. A commercial acrylic-based paint (Roof Seal) was used as the binder. Tween 80 surfactant and deionized water were obtained from laboratory stock.

2.2 Synthesis of CuS nanoparticles

Copper sulfide nanoparticles were synthesized via a co-precipitation method. Briefly, 0.1 mol of copper nitrate trihydrate was dissolved in 200 mL of deionized water and stirred magnetically at room temperature until a homogeneous solution was obtained. Subsequently, 4 mL of diethanolamine and 8 mL of isopropyl alcohol were added under continuous stirring. The pH of the solution was adjusted to 8 using aqueous ammonia. The mixture was then heated to 70 °C and stirred for 30 minutes. Following this, 0.1 mol of thiourea was introduced and dissolved completely. The pH was further adjusted to 10, and the suspension was maintained at 70 °C with vigorous stirring for 2 hours. The resulting black precipitate was collected, washed repeatedly with deionized water, and dried at 60 °C for 24 hours. The final product was ground into a fine powder using an agate mortar and pestle.

2.3 Preparation of CuS–Leucoxene Composite Films

Leucoxene-CuS composite coatings were prepared using Roof Seal as the binder. The detailed procedure is as follows: CuS powder was weighed according to the designated mixing ratios (1:9, 2:8, 3:7, 4:6, and 5:5 CuS-to-leucoxene by weight, as detailed in Table 1). The weighed CuS was finely ground and transferred into a beaker. Then, 1–2 drops of Tween 80 and 5–10 mL of deionized water was added. The suspension was stirred magnetically for 10 minutes, followed by ultrasonication for 15–20 minutes to ensure a homogeneous dispersion. The requisite amount of leucoxene powder and Roof Seal binder were then incorporated. The final mixture was homogenized using a mechanical mixer for 15–30 minutes. The composite slurry was coated onto glass slides using a doctor blade technique to obtain uniform thin films (Fig. 1), which were then air-dried. Two film loading conditions were prepared to evaluate the effect of coating mass on thermal insulation performance. The low-loading condition

refers to a single-layer coating applied using one pass of the doctor blade, yielding an estimated composite loading of approximately 4 mg/cm². The high-loading condition refers to a double-layer coating, in which a second layer was applied after the first had fully air-dried, resulting in an estimated loading of approximately 8 mg/cm². These definitions are used consistently throughout the manuscript.



Fig. 1. Photographic images of nanocomposite films on glass substrates: (a, b) pure Roof Seal, (c, d) pure leucoxene, (e, f) pure CuS, (g, h) 1:9 composite, (i, j) 2:8 composite, (k, l) 3:7 composite, (m, n) 4:6 composite, and (o, p) 5:5 composite.

Table 1. Mixing ratios of copper sulfide and leucoxene composites prepared with Roof Seal binder for coating on glass substrates. The composites were blended with the binder at a fixed proportion of 1 g of composite per 10 mL of binder.

Sample Ratio (CuS:Leucoxene)	Roof Seal (mL)	Roof Seal (g)	Copper Sulfide (g)	Leucoxene (g)
Pure Roof Seal	30	60	0	0
Pure Copper Sulfide	30	60	3	0
Pure Leucoxene	30	60	0	3
1:9	30	60	0.3	2.7
2:8	30	60	0.6	2.4
3:7	30	60	0.9	2.1
4:6	30	60	1.2	1.8
5:5	30	60	1.5	1.5

2.4 Characterization Techniques

The structural and morphological properties of the CuS–leucoxene composites were characterized by X-ray diffraction (XRD, Rigaku SmartLab, Japan) and scanning electron microscopy (SEM). The optical properties were evaluated using UV–Vis–NIR spectroscopy (UH4150, Hitachi, Japan) in diffuse reflectance mode over the wavelength range of 240–2600 nm. Diffuse reflectance spectroscopy (DRS) was employed because the composite films consist of particulate fillers in an acrylic binder, producing rough, scattering surfaces. Unlike specular reflectance (which collects only the mirror-angle reflected beam, appropriate for optically smooth surfaces), DRS uses an integrating sphere to collect all reflected light—both specular and diffuse—at all angles, providing a more representative measurement of the total optical reflectance of paint-type coatings. The elemental composition of leucoxene was analyzed by X-ray fluorescence (XRF).

2.5 Thermal Insulation Measurement

Thermal insulation performance was evaluated by placing the coated glass films on top of a sealed wooden box ($17.5 \times 17.5 \times 32 \text{ cm}^3$) with the coated side facing upward toward the light source. A tungsten lamp (1000 lux) was positioned 20 cm above the film surface to simulate solar IR irradiation. The surface temperature of the coated glass was measured using a thermal imaging camera (HT-02). The internal box temperature was simultaneously recorded using a digital thermometer (Xiaomi Mi) placed inside the box. A K-type thermocouple sensor (1000 mm, banana plug) connected to a digital multimeter (UNI-T UT61B True RMS) was used as an additional internal temperature probe for cross-validation. Measurements were conducted for 40 minutes for all compositions, and for 2 hours for the 5:5 composite to assess steady-state behavior. All experiments were conducted under controlled indoor conditions with the ambient temperature recorded. The temperature difference $\Delta T = T_{\text{surface}} - T_{\text{interior}}$ was used as the primary metric for evaluating thermal insulation efficiency.

3. Result and discussion

3.1 Structural Characterization of Leucoxene–CuS Composites

The morphology of the synthesized CuS nanoparticles was examined using scanning electron microscopy (SEM). As shown in Fig. 2a (20,000 $\times$ magnification), the CuS nanoparticles were predominantly spherical and tended to form agglomerates due to their high surface energy. At higher magnification (50,000 $\times$, Fig. 2b), the nanoparticles exhibited aggregated nanostructures with irregular surface features. In contrast, the leucoxene particles (Fig. 3) displayed irregular to rectangular morphologies with rough surfaces and a broad particle size distribution. Particle agglomeration was also observed, which is typical for naturally derived mineral powders.

The elemental composition of the CuS nanoparticles was confirmed by energy-dispersive X-ray spectroscopy (EDS). The EDS spectrum (Fig. 4) revealed a nearly stoichiometric atomic ratio of Cu:S at 52.2:47.8%, consistent with the stoichiometric composition of CuS.

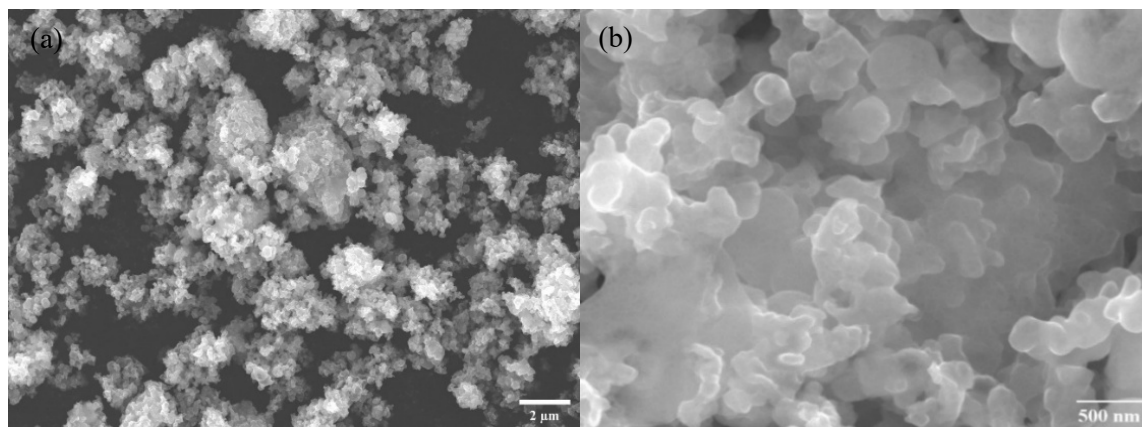


Fig. 2. SEM images of copper sulfide nanoparticles synthesized by co-precipitation: (a) 20,000 $\times$ magnification (scale bar: 2 μm), (b) 50,000 $\times$ magnification (scale bar: 500 nm).

X-ray fluorescence (XRF) analysis was performed to determine the elemental composition of the leucoxene ore. The results, summarized in Table 2, confirmed that TiO_2 was the primary component (87.6%), with minor amounts of SiO_2 (7.0%), Al_2O_3 (1.8%), ZrO_2 (1.15%), and Fe_2O_3 (0.92%). Trace elements accounted for the remaining 1.51%.

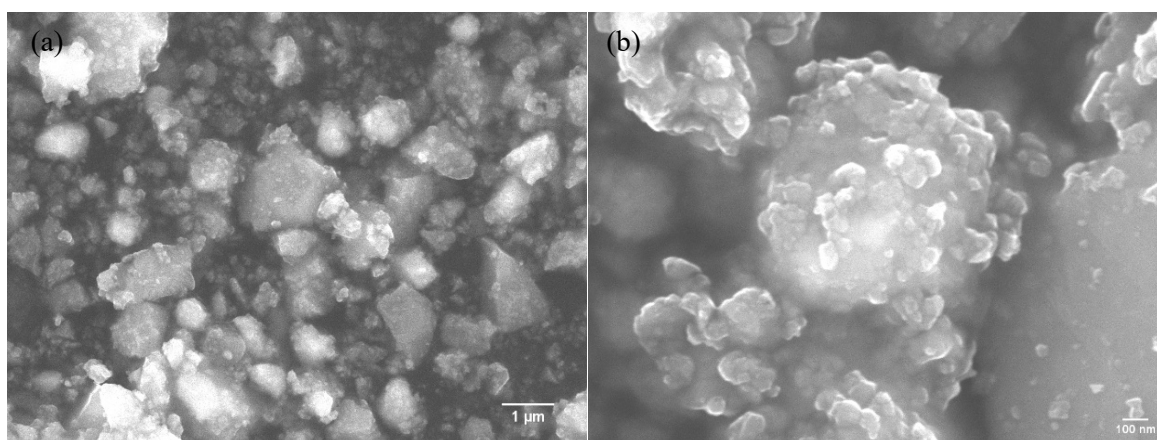


Fig. 3. SEM images of leucoxene particles: (a) 10,000× magnification (scale bar: 1 μm), (b) 50,000× magnification (scale bar: 100 nm).

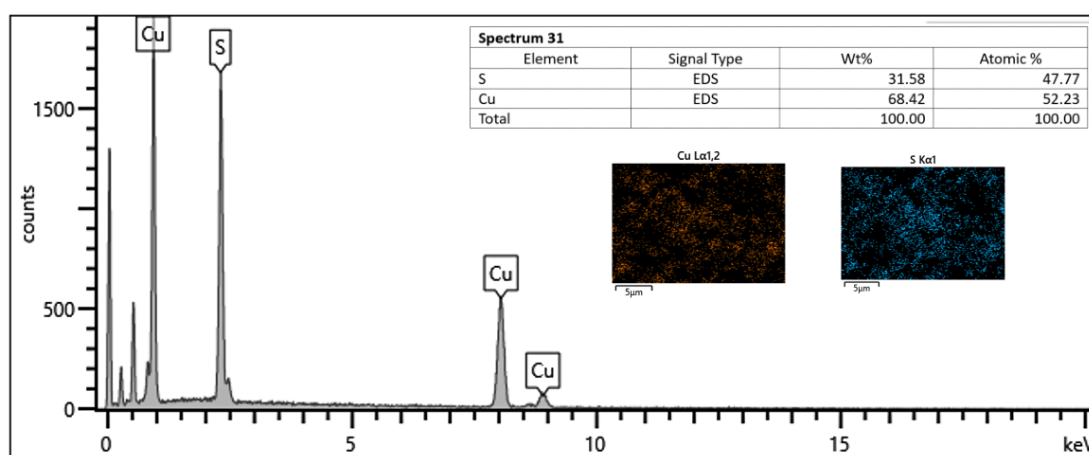


Fig. 4. Energy-dispersive X-ray spectroscopy (EDS) spectrum of the synthesized copper sulfide nanoparticles.

Table 2. Elemental composition of leucoxene ore determined by XRF analysis.

Formula	Concentration (wt%)
TiO_2	87.60%
SiO_2	7.02%
Al_2O_3	1.80%
ZrO_2	1.15%
Fe_2O_3	0.92%
Others	1.51%

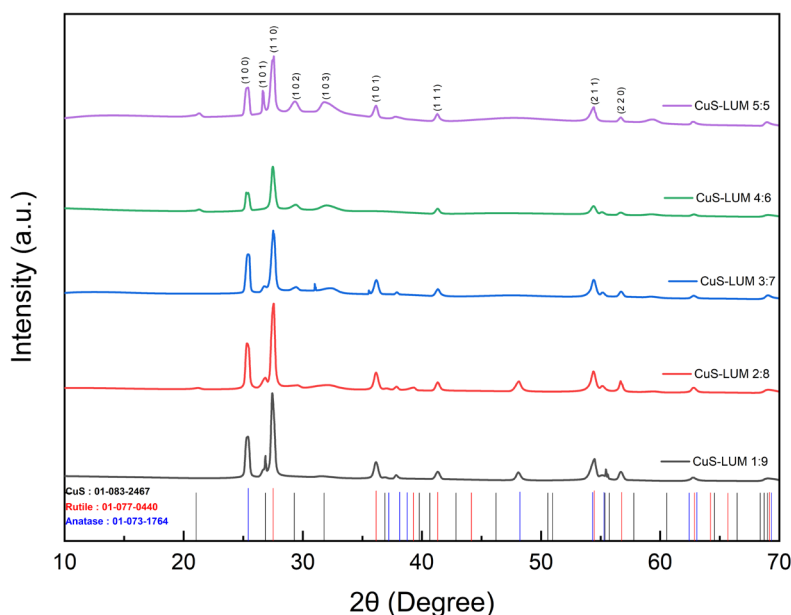


Fig. 5. X-ray diffraction (XRD) patterns of CuS-leucoxene nanocomposites with different CuS-to-leucoxene weight ratios.

The crystalline structures of the CuS–leucoxene composites were analyzed by X-ray diffraction (XRD). The patterns (Fig. 5) confirmed the presence of a hexagonal phase for CuS, with characteristic peaks at $2\theta = 26.86^\circ$, 29.31° , and 31.76° corresponding to the (101), (102), and (103) planes, respectively (ICSD No: 01-083-2467). The leucoxene pattern indicated a mixed phase of TiO_2 , featuring anatase peaks (e.g., 25.37° (101), 48.16° (200); ICSD No: 01-073-1764) and rutile peaks (e.g., 27.44° (110), 36.09° (101), 41.25° (111); ICSD No: 01-077-0440). As the CuS content in the composite increased, the intensity of the CuS diffraction peaks became more pronounced, while the anatase peak intensities diminished, particularly beyond the 3:7 ratio, indicating the growing dominance of the CuS phase in the composite.

3.2 UV-NIR Shielding Properties

The optical properties of the composite films were evaluated using UV–Vis–NIR spectroscopy in diffuse reflectance mode over the 240–2600 nm wavelength range (Fig. 6). The pure Roof Seal film exhibited the highest reflectance ($\sim 85\%$), attributable to its high TiO_2 content. In contrast, the pure CuS film showed the lowest reflectance ($\sim 27\%$) due to its strong absorption across the UV–NIR spectrum. The pure leucoxene film displayed moderate reflectance ($\sim 70\%$). UV Shielding Properties (240–400 nm): In the UV region, the pure leucoxene film exhibited relatively high reflectance ($\sim 60\text{--}70\%$), which can be attributed to the strong UV scattering and absorption characteristics of TiO_2 (the dominant component at 87.6 wt%, Table 2), together with its wide bandgap (anatase: ~ 3.2 eV; rutile: ~ 3.0 eV). In contrast, the pure CuS film showed markedly lower UV reflectance ($\sim 15\text{--}20\%$) due to CuS's strong UV absorption. For the composite films, the UV reflectance decreased progressively with increasing CuS content: the 1:9 composite retained relatively high UV reflectance ($\sim 55\%$), while the 5:5 composite showed the lowest UV reflectance ($\sim 30\%$). This confirms that leucoxene-rich compositions provide superior UV shielding via reflectance, while CuS contributes additional UV absorption. The complementary UV and NIR shielding mechanisms of leucoxene and CuS respectively support the dual UV–IR shielding concept described in the title.

For the composite films, the NIR reflectance systematically decreased with increasing CuS content. The 1:9 and 2:8 composites showed reflectance values of 61% and 60%, respectively. With further increases in CuS loading (3:7, 4:6, and 5:5 ratios), the reflectance progressively decreased to 53%, 50%, and 46%. This trend indicates that increasing CuS content reduces the diffuse reflectance of the composite films, while leucoxene contributes primarily to UV reflection. It should be noted that, for thin-film systems, reduced reflectance could in principle be accompanied by increased transmittance

rather than increased absorption, as governed by $R + T + A = 1$. However, in the present case, the composite coatings incorporate CuS nanoparticles within an opaque acrylic binder matrix. Visual inspection (Fig. 1) confirms the optical opacity of the films, particularly at higher CuS loadings, indicating that transmittance is negligible and that the observed reduction in diffuse reflectance with increasing CuS content may result from a combination of absorption, scattering, reflection, and potentially limited transmission within the composite coating. Therefore, the present diffuse reflectance measurements alone do not allow these individual optical processes to be quantitatively distinguished. The thermal measurements are consistent with the observed optical behavior; however, they do not independently distinguish the relative contributions of absorption, reflection, scattering, and transmission.

Comparing the two loading conditions (Fig. 6a - 6b), the high-loading films showed systematically lower reflectance values than the low-loading films at equivalent CuS:leucoxene ratios. For example, the 5:5 composite showed reflectance of $\sim 46\%$ at low loading versus $\sim 38\%$ at high loading. This is consistent with a greater optical path length and increased absorption by CuS in the thicker double-layer films. Conversely, in the UV region, leucoxene-rich compositions (1:9, 2:8) maintained relatively high reflectance in both loading conditions, confirming the dominant role of leucoxene in UV shielding regardless of film mass.

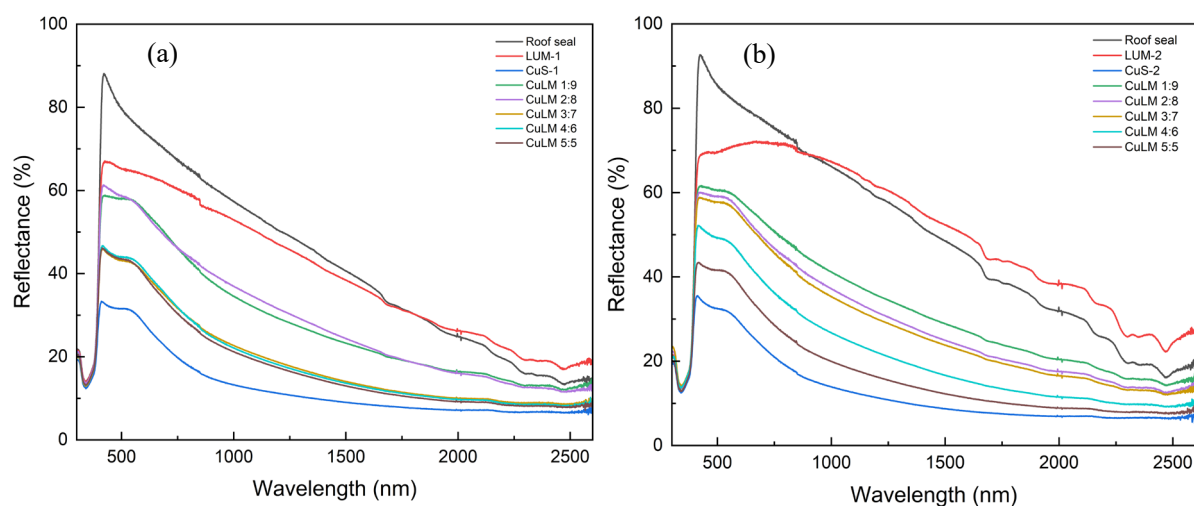


Fig. 6. Diffuse reflectance spectra of CuS–leucoxene composite coatings under two loading conditions: (a) low-loading single-layer films ($\sim 4 \text{ mg cm}^{-2}$, Series 1) and (b) high-loading double-layer films ($\sim 8 \text{ mg cm}^{-2}$, Series 2).

3.3 Thermal Insulation Properties

The thermal insulation performance was evaluated using glass films coated with CuS–leucoxene composites prepared with Roof Seal as a binder. After drying, the coated films were irradiated with infrared light from a tungsten lamp at an intensity of 1000 lux to simulate solar radiation. The experimental setup is described in detail in Section 2.5. Figure 7 shows the temperature evolution inside the closed box and on the film surface under low-loading (single-layer, $\sim 4 \text{ mg/cm}^2$, panels a and b) and high-loading (double-layer, $\sim 8 \text{ mg/cm}^2$, panels c and d) conditions for all CuS:leucoxene ratios. In both loading conditions, temperature increased rapidly upon irradiation and reached a near-steady state within ~ 30 – 40 minutes. For the low-loading films, the internal box temperature increased with increasing CuS content. This behavior is consistent with greater surface heating under IR irradiation. However, the measured temperature response may also be influenced by multiple heat-transfer mechanisms, including heat conduction through the glass substrate, heat accumulation within the coating layer, thermal re-radiation, and natural convection inside the enclosed box. The 1:9 composite exhibited the lowest internal temperature, followed by 2:8, 3:7, 4:6, and 5:5, respectively. The temperature difference ΔT was 40, 40, 39, 41, and 46 $^{\circ}\text{C}$ for ratios 1:9, 2:8, 3:7, 4:6, and 5:5, respectively. For the high-loading films (Fig. 7c, 7d), the same compositional trend was observed, with ΔT values of 28.2, 37.6, 38.7, 40.5,

and 47.5 °C for ratios 1:9, 2:8, 3:7, 4:6, and 5:5, respectively. The temperature difference was used as the primary figure of merit because it represents the thermal gradient across the coated glass system under irradiation. A larger ΔT indicates that a greater proportion of the absorbed heat remains localized at the coating surface rather than being transferred into the enclosed space. Ambient temperature was not used as the reference because the objective of this experiment was to evaluate heat transfer through the coated glass under non-isothermal conditions within the closed-box system. The observed surface heating is consistent with the optical behavior of CuS-containing coatings under IR irradiation. However, the present measurements do not allow the individual contributions of IR absorption, heat conduction, thermal re-radiation, and convective heat transfer to be distinguished.

These results indicate that the 5:5 composition produced the largest surface-to-interior temperature difference under the present experimental conditions. The overall thermal behavior depends on factors such as film thickness, particle dispersion of CuS and leucosene, and the binder characteristics.

As shown in Figure 8, the temperature evolution inside the box and on the film surface was tracked for the 5:5 composite at both loading scales over 2 hours of IR irradiation. The internal temperature stabilized at 36.5 °C (low-loading) and 35.8 °C (high-loading) after 2 h. The surface temperature reached 82 °C and 84.1 °C under the same conditions, giving ΔT values of 45.5 °C and 48.3 °C, respectively. These results indicate an average ΔT of approximately 47 °C for the 5:5 composite, representing the highest surface-to-interior temperature difference among all compositions tested.

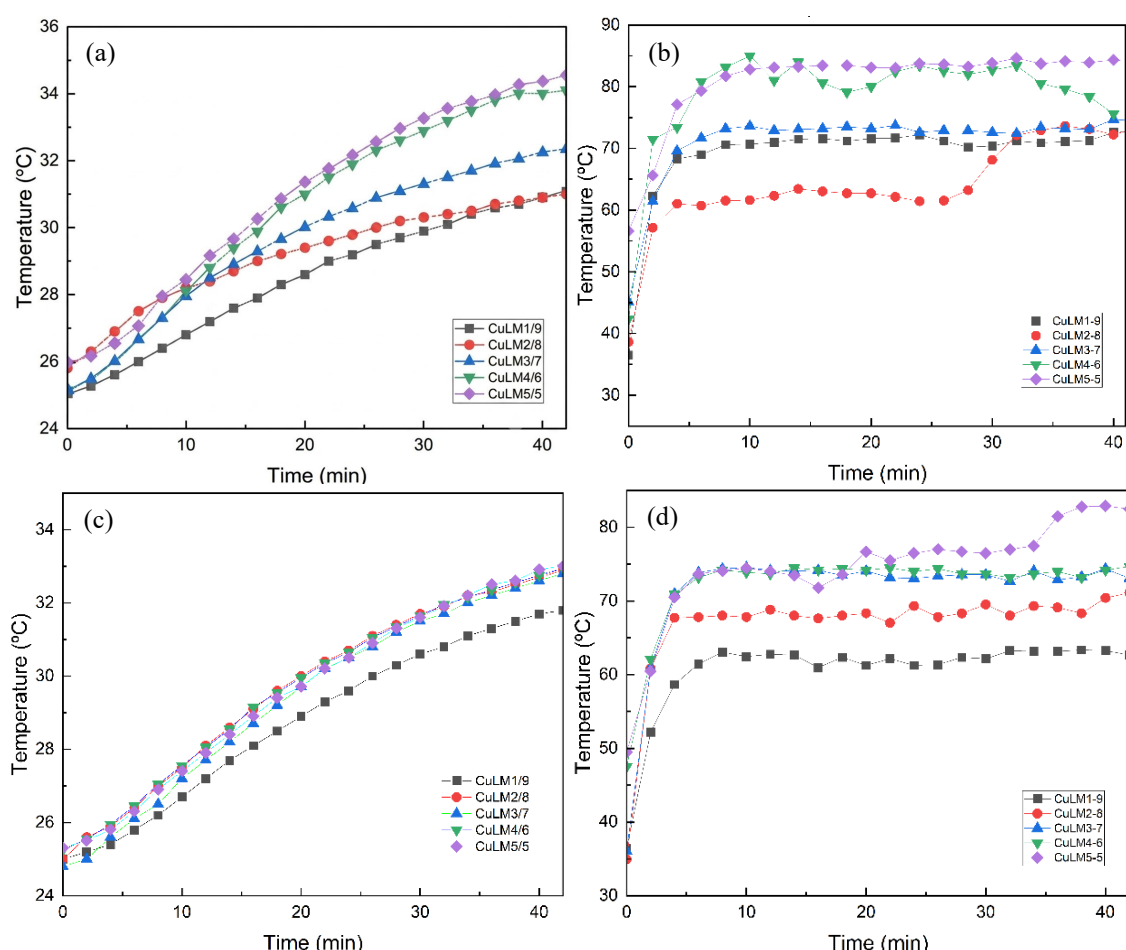


Fig. 7. Temperature evolution of CuS–leucosene composite coatings under IR irradiation: internal box temperature for low-loading and high-loading films in (a) and (c), respectively, and corresponding film surface temperatures in (b) and (d).

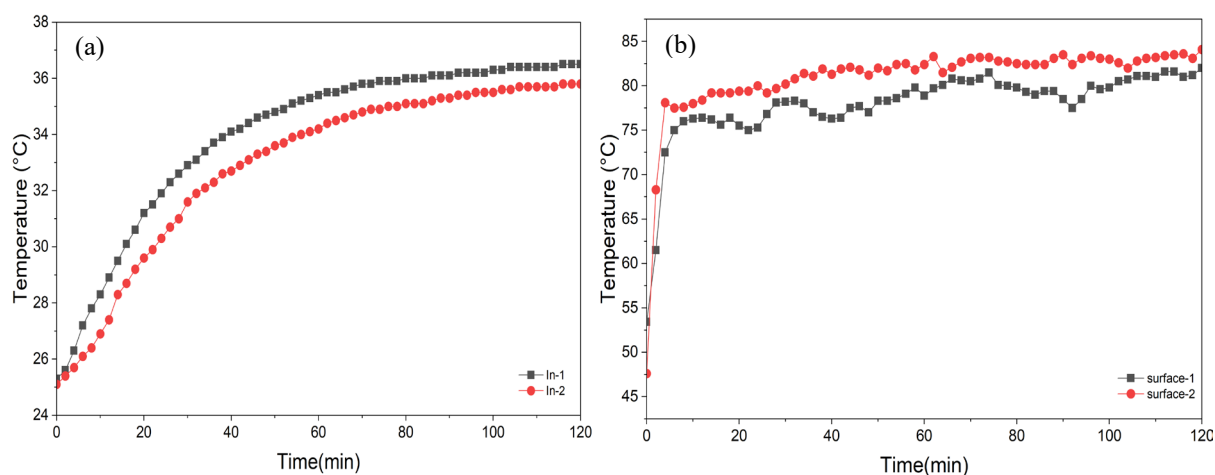


Fig. 8. Temperature profiles for the 5:5 CuS:Leucoxene composite under different loading conditions during 2 hours of IR irradiation: (a) internal box temperature and (b) film surface temperature.

4. Conclusion

In this study, CuS nanoparticles were successfully synthesized via a co-precipitation method and incorporated with leucoxene mineral powder into acrylic-based composite coatings at five CuS-to-leucoxene weight ratios (1:9 to 5:5). XRD analysis confirmed the hexagonal covellite phase of CuS and the mixed anatase–rutile phase of TiO₂ in leucoxene, while SEM revealed spherical CuS nanoparticles (~257 nm) and rectangular leucoxene particles (~235 nm). UV–Vis–NIR diffuse reflectance spectroscopy (240–2600 nm) demonstrated that increasing CuS content progressively reduced diffuse reflectance across both the UV and NIR regions. Conversely, leucoxene-rich compositions maintained higher reflectance in the UV region (240–400 nm), indicating complementary optical responses in the UV and NIR regions. Thermal insulation tests under simulated IR irradiation (1000 lux tungsten lamp) showed that the 5:5 CuS:leucoxene composite at high loading (~8 mg/cm²) exhibited the largest measured temperature gradient ($\Delta T \approx 47\text{--}48\text{ }^{\circ}\text{C}$) between the film surface and the box interior. Importantly, the 1:9 composition achieved comparable thermal performance at substantially lower filler loading, suggesting a cost-effective alternative for practical applications. These findings demonstrate that leucoxene–CuS composites offer tunable optical responses to solar radiation and hold strong promise for energy-efficient window and building envelope applications.

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