

## Enriched huntite as a green flame retardant: thermal and physical behaviors in polyurethane matrices

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**Abstract:** Flame retardants are crucial for operational safety in strategic sectors such as aerospace, automotive, and electronics. However, traditional flame retardants pose significant environmental and health risks due to toxic gas emissions. Consequently, sustainable "green" alternatives like huntite have gained increasing attention. Huntite enhances the thermal stability of polymers through endothermic decomposition, releasing inert gases that suppress combustion without generating toxic byproducts. Polyurethanes (PU) are highly valued in industry for their mechanical strength and flexibility; however, their susceptibility to thermal degradation and structural instability at elevated temperatures restricts their broader application. To meet strict fire safety and performance standards, the development of heat-resistant, multifunctional PU composites is essential. This study investigates the interaction between the polyurethane matrix and the huntite mineral to evaluate its potential as an eco-friendly flame-retardant system. The structural interactions, dispersion behavior, and chemical properties of the resulting PU-huntite composites were systematically characterized using X-ray diffraction (XRD) and Fourier-transform infrared (FTIR) spectroscopy. Flotation-enriched huntite (85.9% purity,  $d_{80} = 46.86 \mu\text{m}$ ) was successfully integrated into polyurethane films (optimized thickness of  $200 \mu\text{m}$ ) at loadings of 0.1, 0.3, 0.5, 1.0, and 3.0 wt.%. XRD analysis revealed the retention of the broad amorphous halo ( $2\theta = 10^\circ\text{--}25^\circ$ ) along with the emergence of the characteristic huntite reflection at  $2\theta = 31.8^\circ$  at 3.0% loading. FTIR spectroscopy confirmed a purely physical incorporation through the appearance of the carbonate ( $\text{CO}_3^{2-}$ ) band at  $870\text{--}880 \text{ cm}^{-1}$  and overlapping bands at  $1400\text{--}1500 \text{ cm}^{-1}$ , while the stability of the carbonyl ( $\text{C=O}$ ) peak at  $1730\text{--}1740 \text{ cm}^{-1}$  confirmed that the polymer backbone remained unaltered. Furthermore, functional evaluations demonstrated that huntite incorporation substantially enhances the hiding power and opacity of the composite films, with the opacity value reaching 83.3% at the maximum 3.0% mineral loading. These findings validate that huntite not only serves as a promising sustainable flame retardant but also significantly improves the light-blocking performance of polyurethane matrices for advanced structural and protective coating applications.

**Keywords:** huntite, polyurethane, green, composite, flame retardant

### 1. Introduction

Traditional flame retardants, particularly halogenated compounds, have faced significant scrutiny due to environmental and health concerns, including toxic gas emissions during combustion, bioaccumulation, and environmental persistence. To meet industry fire safety standards, these halogenated additives are often incorporated at high concentrations, reaching up to 10% in foams and 30% in electronic plastics (Blum, 2015). However, the potential toxicity risks and the lack of comprehensive biological data for these substances necessitate the development of safer, more efficient alternatives that preserve the material's physical integrity while providing high flame resistance (Feng et al., 2023).

In this context, huntite ( $\text{Mg}_3\text{Ca}(\text{CO}_3)_4$ ) emerges as a promising mineral alternative, characterized by a double carbonate structure comprising calcium and magnesium. The fundamental mechanism of huntite in flame retardant applications is governed by its thermal decomposition behaviour. For inorganic mineral flame retardants, the decomposition temperature determines the effective thermal protection range. While

hydromagnesite decomposes at lower temperatures (220–550 °C) to form an initial barrier by releasing water vapor and carbon dioxide (Haurie et al., 2006), huntite provides essential stability under elevated thermal exposure. Upon heating, huntite decomposes endothermically, releasing carbon dioxide gas and leaving behind an inorganic oxide residue. The endothermic decomposition of huntite initiates at approximately 450 °C and continues to release carbon dioxide up to 750 °C. This process enables its classification as an effective mineral-based filler. The emitted carbon dioxide serves to dilute combustible gases in the surrounding environment. At the same time, the residual oxide forms a protective barrier on the polymer surface, thereby impeding the progression of combustion (Hollingbery & Hull, 2010). Because huntite occurs alongside other carbonate minerals (such as dolomite, hydromagnesite, and magnesite) with similar surface properties, selective separation via flotation is often investigated and used to achieve the required high purity before industrial application (Kangal et al., 2005).

Polyurethanes (PU) are widely used in various industries because of their mechanical strength and flexibility; however, their inherent flammability remains a significant drawback. To mitigate this, researchers have explored incorporating huntite and hydromagnesite as reinforcing fillers to enhance the thermal stability and flame resistance of these composites (Güler, 2017). Huntite-hydromagnesite mixtures have been successfully utilized as flame-retardant fillers across various sectors, including the cable, construction, and polymer industries. For instance, integrating these mineral mixtures into acrylate-based polymer paste coatings for cotton fabrics has been shown to influence functional performance; notably, air permeability may decrease depending on the specific coating formulation and mineral loading ratio (Çamlıbel et al., 2019). Beyond its flame-retardant properties, huntite's efficacy as an inorganic reinforcing filler is well documented. For example, Seki et al. (2013) demonstrated that incorporating 3 wt.% huntite into unsaturated polyester composites provided optimal homogeneous dispersion. This specific loading enhanced both mechanical strength and thermal stability, raising the maximum decomposition temperature by 15 °C. Conversely, higher filler contents (e.g., 5 wt.%) induced severe particle agglomeration and void formation, which drastically deteriorated mechanical performance. These findings highlight the need to optimize mineral filler concentrations to improve thermal resistance without compromising the structural integrity of the polymer matrix.

Furthermore, the literature indicates that the flame-retardant efficacy of these systems depends on multiple factors beyond the additive type. Specifically, parameters such as particle size, purity, and mineral homogeneity within the polymer matrix play critical roles in determining the composite's overall performance (Yüçetürk et al., 2023). Filler particle size fundamentally dictates composite performance. Yılmaz Atay and Çelik (2012) demonstrated that nanoscale (0.1 µm) huntite/hydromagnesite additions consolidate the polymer matrix, enhancing its overall properties. Thermally, these minerals decompose (220–600 °C) to release CO<sub>2</sub> and form a protective ceramic barrier that restricts oxygen and suppresses smoke. Thus, precise particle size control is essential for designing effective multifunctional green composites. Effective pretreatment and enrichment strategies are essential to tailor the mineral's particle size and ensure its homogeneous distribution, both of which directly influence the performance and quality of the final product (Türk et al., 2024). Beyond structural reinforcement, incorporating highly white and refractive mineral fillers imparts critical optical functionalities to the polymer. In coating applications, high opacity and hiding power not only completely conceal the underlying substrate but also effectively scatter visible light and radiant heat, providing an additional physical defense mechanism against fire-induced thermal radiation (Wypych, 2016). Therefore, this study incorporated enriched huntite into polyurethane matrices at different loading ratios to assess its compatibility with the polymer and its potential as a sustainable multifunctional filler. The effects of huntite addition on the structural and chemical characteristics of the resulting composite films were evaluated using X-ray diffraction (XRD) and Fourier-transform infrared (FTIR) spectroscopy. In addition, film thickness was optimized, and the influence of huntite content on the opacity and hiding power of the polyurethane films was examined.

## 2. Materials and methods

### 2.1. Materials

The raw material was originally sourced from Çameli/Denizli, Türkiye. However, the sample used in this study is not run-of-mine ore, but a flotation concentrate derived from previous studies. The flotation

concentrate was recovered through a five-stage flotation process at natural pH ( $\sim 8$ ), using 1200 g/Mg potassium oleate (K-oleate) as the collector and 2000 g/Mg sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) as the depressant. The float product, produced under optimum flotation conditions, was characterized using an Epsilon 4 X-ray fluorescence (XRF) instrument. The XRF results indicate that the sample contains 13.49% CaO and 34.76% MgO, corresponding to an MgO/CaO ratio of 2.58. Additionally, the loss on ignition (LOI) was 47.71%.

To assess the purity of the enriched sample, mineralogical characterization was conducted via X-ray diffraction (XRD) using a PANalytical X'Pert Pro diffractometer equipped with a Cu X-ray source. Mineral identification was performed using the ICDD PDF-4/Mineral database, and the relative mineral proportions were quantitatively determined using the Rietveld refinement method. Fig. 1 presents the corresponding XRD pattern.

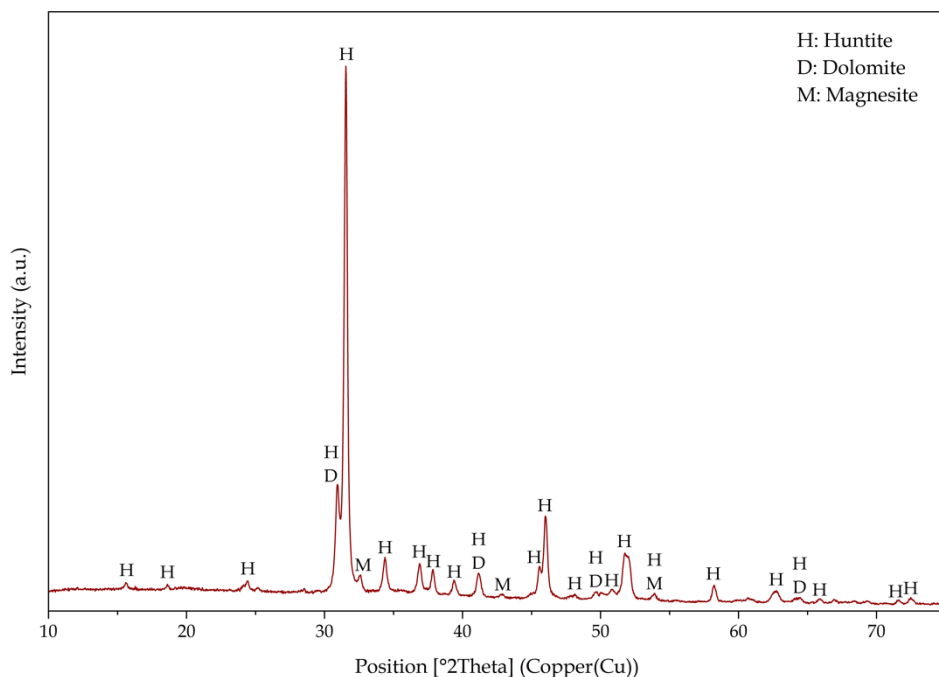


Fig. 1. XRD analysis of the enriched sample

The XRD results demonstrate that the enriched sample consists of approximately 85.9% huntite, 13.1% dolomite, and 1% magnesite. Furthermore, because particle size distribution is a critical parameter for the integration of the huntite sample with polymers, this property was measured using a Malvern 2000 instrument. The results of this analysis are shown in Fig. 2.

According to the particle size distribution curve, the sample exhibits a very fine particle size, with 80% of the material measuring below 50  $\mu\text{m}$ . Specifically, the  $d_{10}$ ,  $d_{20}$ ,  $d_{50}$ ,  $d_{80}$ , and  $d_{90}$  values of the sample were determined as 0.842, 1.463, 5.168, 46.858, and 76.193  $\mu\text{m}$ , respectively. Because huntite typically exhibits a fine-grained nature, the resulting flotation concentrate has a very fine particle-size distribution, further supporting that the material mainly consists of huntite.

Commercial-grade thermoplastic polyurethane (TPU), supplied as pellets, was obtained from Kimpur-Kimteks Polyurethane Inc. (Türkiye) and used as received. After dissolving the polyurethane beads in a suitable solvent, enriched huntite was added as the mineral filler to prepare the PU-huntite composite films.

## 2.2. Methods

The structural and chemical characteristics of the neat polyurethane (PU) were initially evaluated using X-ray diffraction (XRD) and Fourier-transform infrared (FTIR) spectroscopy. They were subsequently compared with those of the huntite-integrated PU matrices. XRD analyses were performed using the same diffractometer described in the Materials section, while FTIR spectra were recorded at room temperature over the wavenumber range of 500–4000  $\text{cm}^{-1}$  using a Jasco FT/IR-4700 spectrometer.

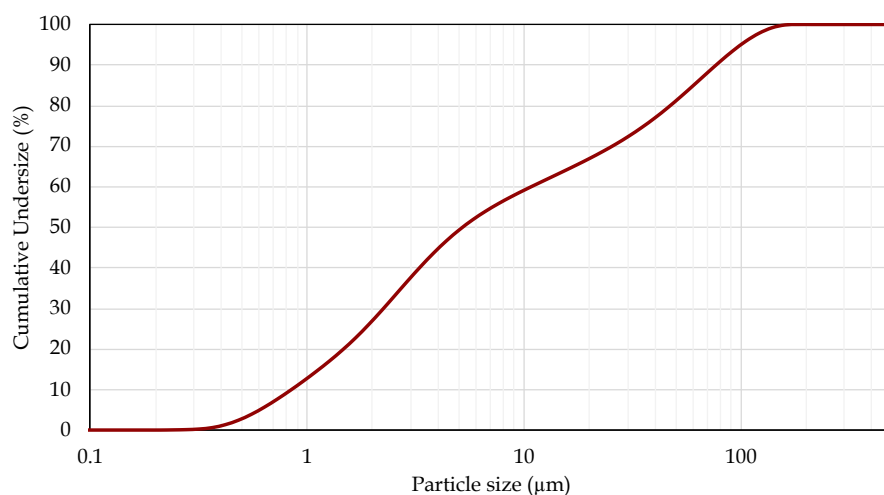


Fig. 2. Particle size distribution of the huntite sample

To formulate the polymer solution, PU beads were dissolved in N-methyl-2-pyrrolidone (NMP) at approximately 10% (w/w) polymer concentration, heated to 60 °C, and stirred overnight at 400 rpm using a magnetic stirrer. Following dissolution, purified huntite was incorporated into the PU matrix at varying mass fractions of 0.1%, 0.3%, 0.5%, 1.0%, and 3.0% (w/w) relative to the polymer content. To ensure the homogeneous dispersion of the mineral filler, the composite mixtures were maintained at 60 °C and stirred for 48 hours using a high-speed mechanical stirrer.

To prepare the PU films, PU solutions of varying thicknesses were applied onto a glass substrate using a BYK four-sided cylindrical film applicator. The film-casting process was performed using a Sheen automatic film application device. Films with thicknesses of 100 μm and 200 μm were prepared and then placed in a water bath coagulation process. The cast films were subsequently immersed in a distilled-water coagulation bath at room temperature, where solvent-nonsolvent exchange induced nonsolvent-induced phase separation (NIPS) and film solidification. The general composite film preparation approach was adapted from Benli (2013), while the film casting and aqueous coagulation procedure was conducted according to the conventional nonsolvent-induced phase separation approach. After coagulation, flexible, matte-white films were successfully obtained.

After examining the behavior of the PU-huntite matrixes, the opacity of the films containing different huntite loadings was measured using an X-Rite SP64 Portable Sphere Spectrophotometer. The opacity values were calculated according to the method specified in the TS EN ISO 6504-3 standard.

### 3. Results and discussion

The incorporation of huntite was limited to a maximum of 3 wt.% in the PU-huntite composites. Beyond this loading, huntite's extremely fine particle size and high surface area caused significant mixing resistance and dispersion difficulties, driven by particle agglomeration and poor matrix wetting. Because the neat polyurethane solution was already highly viscous, adding higher amounts of huntite significantly hindered the stirring process and resulted in a heterogeneous mixture. Consequently, a 3% huntite loading was determined to be the upper limit. Fig. 3 presents the successfully prepared homogeneous PU-huntite composite mixtures.

The thickness of the PU films is a crucial factor because it influences the polymer's mechanical stability. Initially, PU films were applied onto glass substrates at thicknesses of 100 μm and 200 μm using a cylindrical film applicator. As shown in Fig. 4, the 200 μm films showed better mechanical stability and flexibility than the 100 μm samples. Therefore, a thickness of 200 μm was selected as the standard parameter for producing all subsequent composite films in this study.

#### 3.1. XRD analysis

To characterize the materials, both the neat PU and the prepared huntite-polymer composites were first subjected to X-ray diffraction (XRD) analysis. The analysis was performed on a PANalytical X'Pert Pro

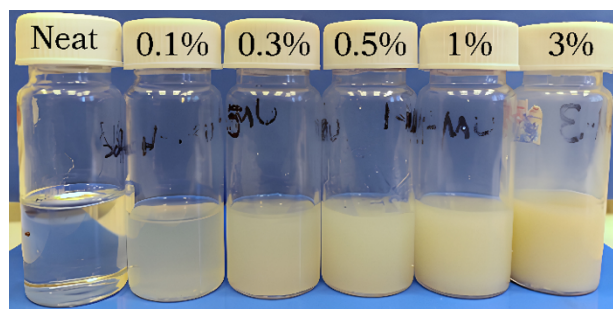


Fig. 3. Neat PU and PU-huntite composites



Fig. 4. Neat PU films: (a) 100  $\mu\text{m}$  and (b) 200  $\mu\text{m}$

diffractometer equipped with a Cu X-ray source (Cu K $\alpha$ ,  $\lambda = 1.5406 \text{ \AA}$ ) at an operating voltage of 45 kV and a current of 40 mA. Data were collected in the  $2\theta$  range of  $5^\circ$ – $70^\circ$  with a step size of  $0.02^\circ$ , and phase identification was evaluated via PANalytical HighScore software using the ICDD PDF-4/Minerals database. XRD analysis allows a comprehensive evaluation of the internal structural arrangement and crystalline properties of the polymeric composite, facilitating determination of whether the incorporated additives induce significant structural changes within the matrix. All XRD measurements were performed on the neat polymer and the composites, all with a film thickness of 200  $\mu\text{m}$ . The resulting XRD patterns are given in Fig. 5.

The XRD patterns show how the polymer structure changes when huntite is added at 0.1% to 3% by weight. The pattern for the neat polymer shows a broad, low-intensity feature between  $10^\circ$  and  $25^\circ$ . This broad feature is characteristic of the disordered, amorphous regions of the polyurethane. A very sharp, intense peak also appears around  $44^\circ$ , with a smaller one near  $64^\circ$ . These sharp peaks indicate that the neat polymer also contains highly ordered crystalline regions.

When small amounts of huntite (0.1%, 0.3%, 0.5%, and 1%) are added, the patterns remain almost similar to those of the neat polymer. The broad feature and the sharp peak at  $44^\circ$  do not change. At these low amounts, the characteristic huntite peaks are not clearly distinguishable. The limit of detection (LOD) for crystalline phases within amorphous or complex matrices typically ranges between 0.1 and 1 wt.% using standard laboratory diffractometers. Because amorphous materials lack crystalline order, they produce a broad amorphous halo and elevated background noise in XRD patterns (Jendrzewska et al., 2022). At low addition ratios, the weak diffraction peaks of huntite crystals are masked by this dominant amorphous background, making identification challenging because the signal-to-noise (S/N) ratio is significantly reduced. This suggests that the huntite particles are either present at concentrations below the instrument's detection limit against the polymer background or are distributed homogeneously within the polymer matrix.

When the maximum amount of huntite (3%) is added, a new, clear peak appears around  $31^\circ$ . This finding strongly agrees with the primary crystallographic peak of the huntite mineral, which occurs at  $31.8^\circ$  as specified in the ICDD PDF-4 database (Card No. 14-0409) and reported in previous studies

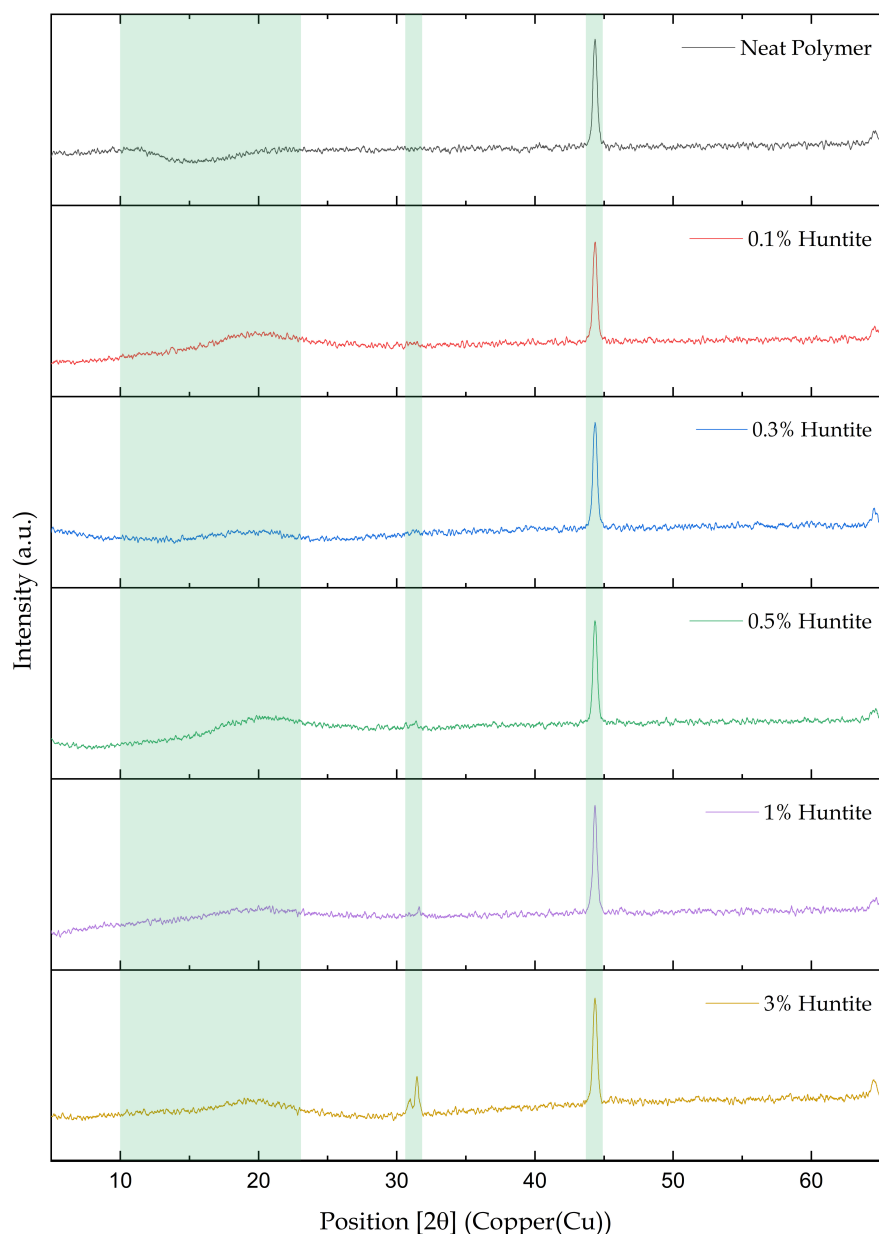


Fig. 5. XRD patterns of neat PU and PU-huntite composites

(Türk et al., 2024; Keli, 2021; Hollingbery and Hull, 2010). As shown in the XRD patterns (Fig. 5), progressively incorporating huntite (0.1 to 3 wt.%) promotes a broad amorphous halo between  $10^\circ$  and  $25^\circ$  ( $2\theta$ ). When huntite particles are mixed with the polymer, they become physically dispersed among the polymer chains. As the huntite particles distribute within the polymer matrix, the spacing and physical interactions among the polymer chains are altered. This physical separation and steric hindrance disrupt the close packing and ordered alignment of polymer segments, increasing the scattering intensity of the amorphous phase. Rather than indicating structural degradation or phase instability, this behavior reflects filler-induced suppression of matrix crystallization (amorphization). According to the literature, incorporating inorganic mineral fillers interferes with the formation and compaction of the polymer's rigid segments, hindering localized crystallization and consequently reducing the overall crystallinity index of the composite system (Alves et al., 2022). The systematic broadening of this amorphous feature confirms homogeneous integration and physically stable dispersion of the mineral phase within the composite matrix. The wide curve and the sharp  $44^\circ$  peak of the polymer remain unchanged. Overall, these XRD patterns indicate that mixing huntite into the polymer is primarily a physical process. Huntite does not damage or alter the polymer's original

structure, and the mineral retains its native crystalline structure without causing major structural changes.

### 3.2. FTIR analysis

After determining the crystalline structure of PU-huntite composites, the composites were subjected to Fourier-transform infrared (FTIR) spectroscopy. FTIR spectroscopy was used to identify specific functional groups and verify the chemical structure of the polyurethane. Fig. 6 shows the FTIR spectra of neat PU and PU-huntite composites.

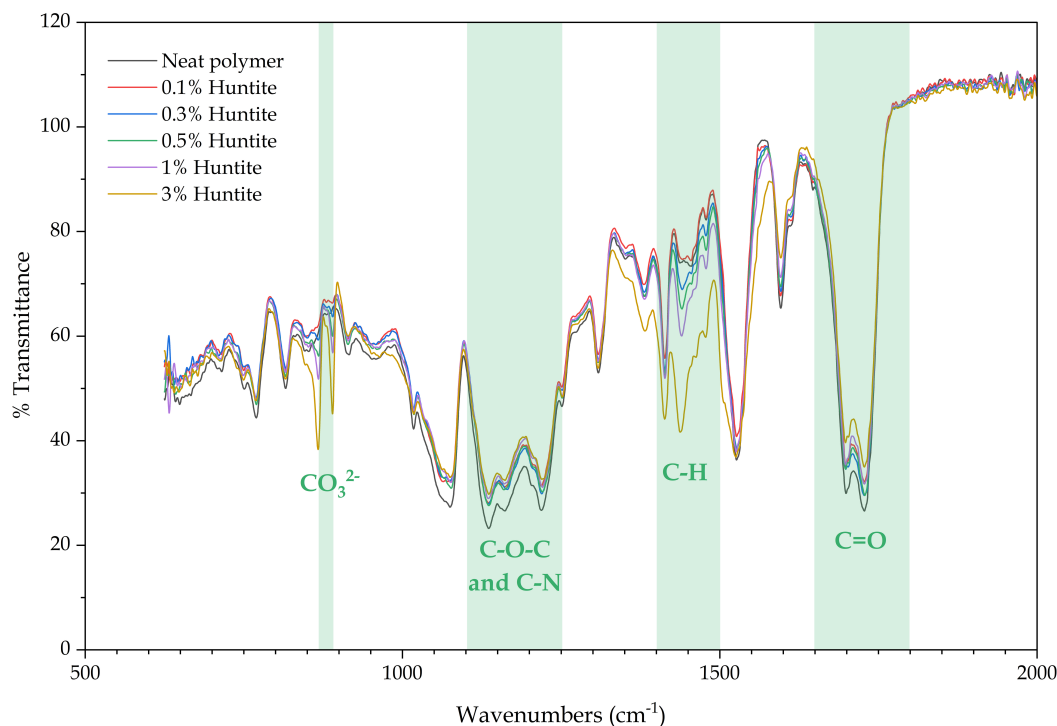


Fig. 6. FTIR spectra of neat PU and PU-huntite composites

The Fourier-transform infrared (FTIR) spectra show the chemical signatures of the neat polyurethane (PU) matrix and the PU-huntite composites in the 500-2000  $\text{cm}^{-1}$  wavenumber range. The provided FTIR spectral data indicate the nature of the interaction between the polymer matrix and the huntite mineral, as well as the significance of the observed changes.

The spectra show no new bands attributable to covalent cross-links or carbon-metal bonds. Instead, they display a superposition of the polymer's inherent peaks and the characteristic peaks of the huntite mineral. This indicates that huntite, an inorganic carbonate mineral, does not form covalent chemical bonds with the polymer matrix. The interaction relies on physical and secondary forces. Polar groups within the polymer, such as carbonyl and ester groups, interact with polar carbonate groups on the surface of the huntite particles through dipole-dipole interactions and van der Waals forces. Consequently, the mineral is physically entrapped within the polymer chains without altering the system's fundamental chemistry.

Regarding the specific wavenumber ranges, a decrease in transmittance on the Y-axis signifies an increase in the absorption of infrared light, which corresponds to a higher concentration of the vibrating bonds. The deepening observed in the 870-880  $\text{cm}^{-1}$  range indicates huntite concentration. While the neat polymer shows a minimal signal in this region, adding 3% huntite creates a prominent peak corresponding to the bending vibration of the carbonate ion, serving as a clear marker confirming mineral incorporation.

Similarly, the broadening and deepening of the curve between 1400 and 1500  $\text{cm}^{-1}$  represents the overlap between the polymer's C-H bending vibrations and the asymmetric stretching vibrations of the carbonate groups in huntite. Because carbonate bonds possess a high dipole moment, they absorb

infrared radiation strongly. Signal amplification in this region indicates that the inorganic particles are dispersed among the polymer chains, with the inorganic signals overlapping the organic ones.

Furthermore, examining the general baseline of the curves reveals a downward shift between 1000 and 1500  $\text{cm}^{-1}$  in spectra with higher huntite contents. This downward shift indicates a physical and optical effect. As solid inorganic mineral powders are introduced into the polymer, the material becomes opaque. These dispersed particles scatter light, reducing the infrared light that passes through the sample and reaches the detector, which appears as a broad drop in overall transmittance.

Finally, the carbonyl peak located between 1730 and 1740  $\text{cm}^{-1}$  remains at the same position across all samples, with no observable frequency shift. This shows that adding the huntite mineral does not chemically alter the polymer matrix backbone. The ester and carbonyl structures remain chemically intact, confirming that the composite is a physical mixture.

### 3.3. Opacity measurements

After confirming structural and chemical integrity via XRD and FTIR analyses, the hiding power of the composite films was evaluated. In flame-retardant coatings, hiding power is a critical parameter, as enhanced opacity improves the thermal shielding efficiency against radiant heat while ensuring complete surface protection of the substrate. PU-huntite composites were applied to black and white panels to measure their hiding power. Specifically, the opacity of the PU-huntite composites was measured to determine the effect of the mineral filler on light transmission. All measurements were performed in triplicate, and the mean values were utilized for the calculations. These calculations were conducted according to Eq. 1, as specified in the TS EN ISO 6504-3 standard methodology. The resulting opacity values are presented in Fig. 7.

$$H_{10} = \frac{Y_{10b}}{Y_{10w}} \cdot 10^2 \quad (1)$$

where  $H_{10}$  is the hiding power,  $Y_{10b}$  is the mean value of the tristimulus value measured over the black areas, and  $Y_{10w}$  is the mean value of the tristimulus value measured over the white areas.

The plotted data exhibit a non-linear polynomial trend. While opacity values vary initially at lower filler fractions (0.1% to 1%), a substantial and sharp increase is observed as the huntite content rises to the maximum loading of 3%, where the hiding power reaches 83.3%. This trend shows that adding huntite, particularly at higher concentrations, effectively reduces the light transmittance of the polyurethane matrix, giving the films a highly opaque structure.

This notable increase in opacity is primarily attributed to the inherent high whiteness and superior light-scattering properties of the huntite mineral dispersed within the polymer network. In coating and membrane applications, minimizing the visibility of the underlying substrate and achieving a uniform, homogeneous surface are critical performance criteria. Consequently, the significant hiding power achieved with increasing huntite content directly supports the applicability of these developed composite films in various industrial sectors, including structural coatings, surface-protective polymer

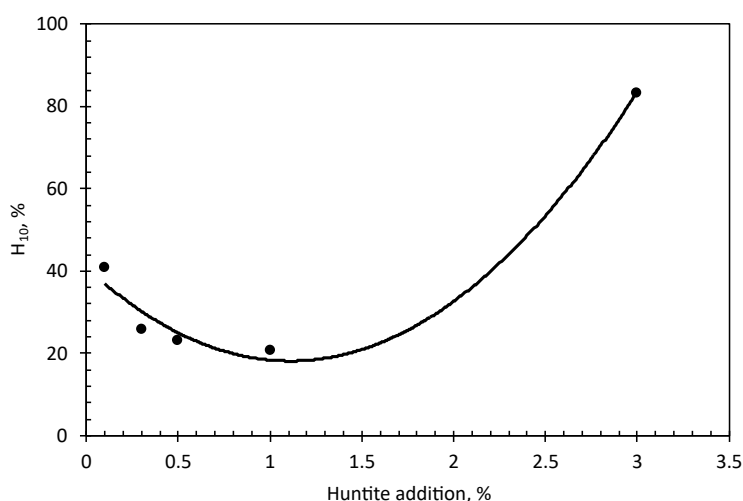


Fig. 7. Hiding power ( $H_{10}$ ) of the PU-huntite composites

layers, and functional membrane systems. Ultimately, these findings confirm that huntite-filled polyurethane formulations can serve as multifunctional material systems, offering highly effective coating performance alongside their established physical stability.

#### 4. Conclusions

This study successfully demonstrates the potential of enriched huntite as a functional and eco-friendly filler within polyurethane matrices. Through experimental evaluation, an optimal film thickness of 200  $\mu\text{m}$  was determined to provide superior mechanical stability and flexibility when applying the polymer solutions onto glass substrates. Because the neat polyurethane solution is highly viscous, the maximum huntite addition was set at 3% by weight to ensure homogeneous dispersion and maintain a workable mixture viscosity. Structural and chemical characterizations by X-ray diffraction and Fourier-transform infrared spectroscopy confirmed that integrating huntite into the polymer network is a purely physical process. The primary semicrystalline structure and the fundamental urethane linkages of the polymer backbone remained entirely intact across all loading ratios. At the same time, the distinctive crystallographic and chemical signatures of the huntite mineral were preserved within the composite matrix.

Furthermore, functional evaluation of the composite films showed that huntite incorporation substantially improved the polyurethane's hiding power. The opacity of the films increased significantly with higher mineral content, reaching 83.3% at a huntite loading of 3%, which is directly attributed to the inherent high whiteness and strong light-scattering properties of the dispersed huntite particles. Ultimately, these results indicate that huntite-filled polyurethane formulations yield a highly versatile, multifunctional material system. By effectively reducing light transmittance and potentially serving as a green flame retardant, huntite makes these composite films promising candidates for advanced industrial applications such as structural coatings, surface-protective layers, and functional membrane systems where both high physical stability and effective substrate concealment are critical performance criteria.

Building on these promising findings, future research should evaluate the thermal degradation behavior of these composites through standardized flammability testing. Additionally, comprehensive mechanical characterization, including tensile testing and substrate adhesion analysis, would provide deeper insights into the physical robustness of the films. Finally, assessing the long-term environmental durability and weathering resistance of these coatings will be essential to fully validate their overall performance and lifespan in practical, real-world applications.

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