

FTIR ANALYSIS OF SOME FLAME RETARDANT AND INTUMESCENT MATERIALS FOR THE IRRIGATION SYSTEMS

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Abstract: The present work aimed to evaluate non-toxic raw materials that will be part of new hybrid and ecological polymer composite materials, used for fireproof and intumescent treatment of systems and equipment associated with watering of cultivated or uncultivated land surfaces, respectively for land irrigation or for extinguishing vegetation fires, as a feasible, sustainable, efficient and economical alternative to synthesized fireproof and intumescent materials. The possible ecological components of the objective hybrid polymer composite material, which were analyzed, are: Alumina, Liquid starch, Powdered starch, Borax, Zinc borate. The results of the analyses were interpreted by correlation, reporting and comparison with the results obtained from established materials in the field of passive fire protection, which have recognized fire resistance performance, such as: Coroparaffin, Naftoblend ACFR-01, NANO PROTECT - NANO-PROTECT® for plastics, Nanomagicplastcare, Nanoprotect pre-cleaning and Antimony trioxide. Liquid PVC was analyzed, with the intention of introducing it as an additive during the preparation of hybrid materials, both to improve the physical and mechanical properties and to facilitate subsequent application even in difficult weather conditions, on site, in the field. The research strategy was to choose the component materials so that the resulting hybrid polymer film would not burn, prevent the spread of fire and smoke, maintain its structural integrity and align with the principles of the circular economy. The substances, possibly components of the objective polymer film, were analyzed by Fourier Transform Infrared Spectroscopy (FTIR), in the “pre-burn” phase, respectively before combustion.

Keywords: fireproof and intumescent coating, FTIR spectrometry, fire retardants.

1. INTRODUCTION

Irrigation of agricultural lands represents one of the most important problems of the contemporary world, being one of the necessary measures to increase harvests and ensure the stability of agricultural production, in dry areas of the country (with a predisposition to desertification), or where precipitation is deficient and unevenly distributed [1,2].

The fireproof and intumescent treatment of the irrigation system or passive fire protection (PFP Passive Fire Protection) represents another important argument, considering the purpose for which they were built, namely intervention in the event of vegetation fires, with the role of maintaining the stability of the structure and limiting the spread of fire/smoke, without active human intervention. The measure is justified by the increasing incidence of fires of combustible plant material (forests, meadows, stubble, bushes), which are expanding rapidly,

endangering human and animal lives, affecting the environment, destroying biodiversity, degrading ecosystems, polluting the air, leading to a decrease in soil fertility, often devastating inhabited areas and destroying infrastructure. In Romania, vegetation fires are a major problem, affecting thousands of hectares annually, with a sharp increase in frequency, with an average of dozens of such fires being recorded daily, especially in spring periods, when farmers resort to clearing land by voluntarily burning vegetation on their land, a fire that often gets out of control [3]. In March 2026, over 600 fires have already consumed 1,500 hectares. [4]. To effectively manage the phenomenon, a complex approach is essential, which includes both measures for monitoring risk areas, prevention through education and specific regulations, and rapid action in case of emergency, through the use of fire-retardant and intumescent treated intervention systems.

In the context of passive fire protection (FR - Flame Retardant), it is usually indicated that the material or system in question has been treated or specially designed to resist combustion and slow the spread of fire. FR technologies are predominantly applied to ecological systems that operate on large areas of land, such as agricultural fields, grazing fields, vineyards, forests, meadows, lakes, deltas, both for irrigation and for fire prevention, crop protection and habitat conservation [5].

2. ANALYTICAL CHARACTERIZATION OF MATERIALS

2.1. General Considerations

Analytical characterization of materials is a fundamental process in the field of materials science without which a scientific understanding of materials engineering could not be established and is defined as the procedure by which the chemical composition, physical structure and properties (mechanical, thermal, surface) of a material are examined and measured, using advanced techniques, in order to understand the relationship between structure and properties. [6,7]. This involves the use of qualitative and quantitative methods for the identification of components. According to different scientific interpretations, the properties of materials can be studied both by macroscopic techniques, microscopic techniques and thermal techniques.

2.2. Chemical characterization of materials

The chemical characterization of materials is carried out by analytical methods that allow the identification of the composition, molecular structure and purity of substances. These are essential to understand the correlation between composition and the final properties of a material.

The choice of method depends on the nature of the sample (organic or inorganic), the required precision and whether major constituents or traces of impurities are being monitored [8-10].

In this scientific research, FTIR spectrometric analysis methods was used for the chemical characterization of materials, namely the identification of elements and chemical bonds based on the interaction with electromagnetic radiation, namely: Fourier Transform Infrared Spectroscopy (FTIR) – which identifies the chemical structure and functional groups, being useful for polymers and insulating materials.

2.3. Properties of fireproof and intumescent materials

Fireproofing and intumescence are structural and application means designed for passive fire protection, respectively to limit the spread of fire, smoke and heat, thus ensuring the stability of a structure for a given time, without actively extinguishing the fire (unlike active systems such as sprinklers) [11]

Although often confused, they function differently: fireproofing reduces the flammability of surfaces, while intumescence acts as an expandable thermal barrier [12]. The main properties of fireproof and intumescent materials were taken into account when comparing the properties of the raw materials used and selecting the optimal ones to achieve the objective of this scientific work [13].

Flame retardant materials (gels, sprays, varnishes, paints, etc.) are designed to be applied to combustible surfaces, such as wood, plastic or textiles, and have the following properties:

- reduce flammability by decreasing the speed of ignition and propagation of flames on surfaces;
- reduce the amount of toxic gases and smoke emission released during combustion;
- suffocates the flame when exposed to high temperatures, by releasing substances (e.g. ammonium phosphates);
- also provide protection against oxidative, corrosive factors, etc.;
- are easily applied by immersion, spraying or brushing on structural materials.

Intumescent materials (paints, mastics, gels, varnishes, etc.) are specially designed to react to extreme heat, being used especially for the structural protection of steel, plastic and wood and have the following properties [14,15]:

- they produce the expansion effect upon exposure to heat (at high temperatures usually above 200°C - 300°C), respectively the material swells, increasing its thickness up to 50 times.
- they form a thermal insulation barrier, respectively a layer of carbonized foam (char) that isolates the supporting material (steel, wood, plastic) from high temperatures, maintaining its structural integrity.
- they provide long-term protection, respectively they maintain the load-bearing capacity of the building, providing valuable time for evacuation (from 30 to 120+ minutes).
- they are non-toxic (in normal conditions), most modern intumescent paints being water-based, environmentally friendly and do not emit harmful gases when applied.
- they do not modify the mechanical properties, their application not modifying or altering the structural resistance of the protected material.

3. EXPERIMENTAL RESULTS

The FTIR spectrum of alumina (Fig.1) confirms its flame retardant properties before combustion by the presence of specific characteristics (especially aluminum trihydrate - ATH or aluminum hydroxide) related to the hydroxyl groups (-OH) and Al-O bonds, which are responsible for its flame retardant (endotherm) action at high

temperatures ($900\text{--}1100\text{ cm}^{-1}$ (Al-O-H Deformation) and the $400\text{--}900\text{ cm}^{-1}$ region (Al-O Stretch/Deformation).

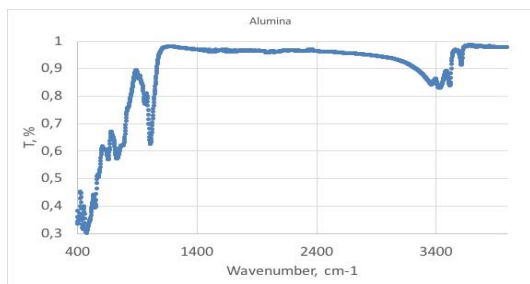


Figure 1. FTIR Spectrum for Alumina

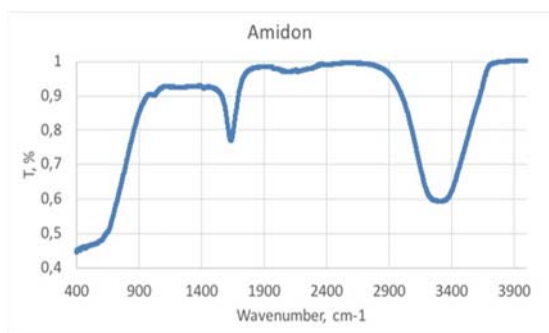


Figure 2. FTIR Spectrum for Liquid Starch

The FTIR spectrum of liquid (gelatinized) starch (Fig.2) intended for flame retardant and intumescent applications, *prior to combustion*, exhibits specific characteristics that indicate the structure of the starch (as a carbon source): Region $900\text{--}1500\text{ cm}^{-1}$ (Fingerprint Starch/C-O-C Bonds), assigned to the crystalline /amorphous starch structure, and Glycosidic Bonds (C-O-C) in the $1000\text{--}1100\text{ cm}^{-1}$ region.

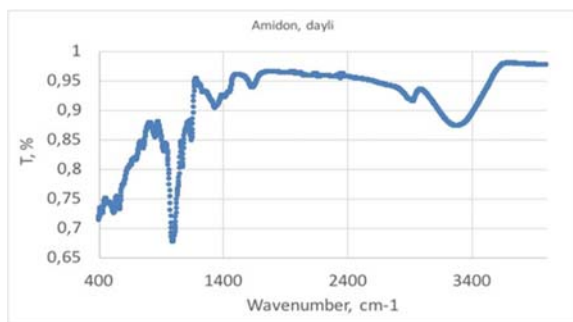


Figure 3. FTIR Spectrum for Starch Powder (Dayli)

Before combustion, the spectrum does not show carbonization products, i.e. there are no strong bands of aromatic C=C or C=O typical of graphite carbon. It only demonstrates the structural integrity of the starch and the physical presence of the components, without presenting the aromatic carbon structures (char) that occur only

during combustion, and which will form an insulating layer by decomposition, esterification and gas release, a process monitored by decreasing the intensity of the -OH and -CH bands and the appearance of carbonization bands at high temperatures. In the region of the "Fingerprint" ($900\text{--}1200\text{ cm}^{-1}$), the distinct bands around 990 , 1020 and 1050 cm^{-1} are highlighted and are important for analyzing the crystalline/amorphous structure of starch.

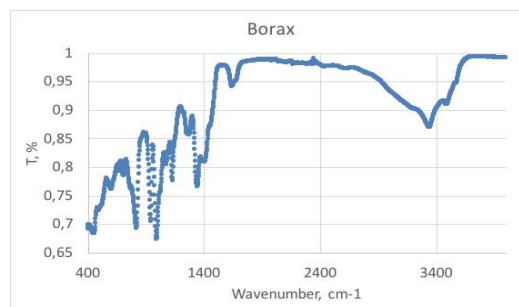


Figure 4. FTIR spectrum for Borax

FTIR spectroscopy applied to borax (sodium tetraborate decahydrate - $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) (Fig.4) prior to combustion, in the context of flame retardancy and intumescent behavior, reveals characteristic bands related to the vibrations of the B-O (boron-oxygen), OH (hydroxyl) bonds and the presence of crystallization water. Borax works as a flame retardant by releasing crystallization water to heat and forming a glassy layer (char) that insulates the material. The main evidences are: Region $1400\text{--}1500\text{ cm}^{-1}$ (vibration B-O trigonal): Strong bands are highlighted, corresponding to the vibrations of asymmetrical stretching of the BO_3 units (trigonal borons), Region $800\text{--}1400\text{ cm}^{-1}$ (Tetrahedral B-O Vibrations): Characteristic bands for the tensile vibrations of the BO_4 (tetrahedral boron) units, essential for the structure of sodium borate, stand out, Region below 700 cm^{-1} (Deformations): Bending bands of boron-oxygen bonds appear.

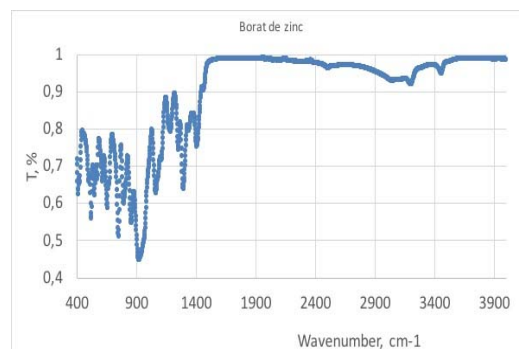


Figure 5. FTIR Spectrum for Zinc Borate

FTIR spectrum of zinc borate (typically $2\text{ZnO} \cdot 3\text{B}_2\text{O}_3 \cdot 3.5\text{H}_2\text{O}$ or $4\text{ZnO} \cdot \text{B}_2\text{O}_3 \cdot \text{H}_2\text{O}$) (Fig. 5) before combustion is characterized by absorption bands

specific to the borate groups and crystallization water, essential for its flame retardant mechanism.

Region 1300–1500 cm^{-1} : Strong bands characteristic of the asymmetrical stretching vibrations of the B-O bonds in the BO_3 units (triangular borate) are highlighted.

Region 900–1200 cm^{-1} : Bands associated with the tensile vibrations of the B-O bonds in the BO_4 (tetrahedral borate) units appear.

Region < 800 cm^{-1} : B-O-B bond deformation bands and Zn-O vibrations (metal-oxygen bonds) are highlighted.

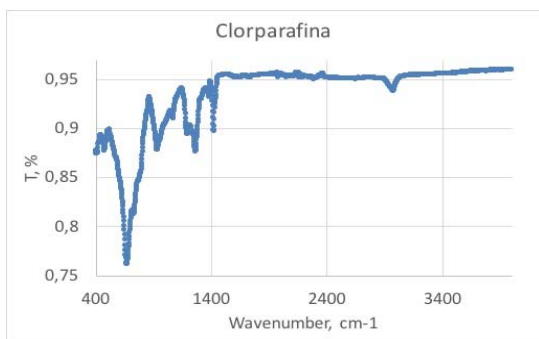


Figure 6. FTIR Spectrum for Chloroparaffin

The absence of significant bands in the area 1600–1700 cm^{-1} , leads to the conclusion of the absence of double bonds ($\text{C}=\text{C}$), which means that it is a high-quality saturated chloroparaffin (Fig.6), and indicates that degradation or dehydrochlorination (HCl removal) has not begun. In intumescent formulas, chloroparaffin acts as a hydrochloric acid (HCl) release agent at high temperatures ($>200^\circ\text{C}$), which acts catalytically to dehydrate the carbon source and form the carbonized layer.

Region 600–1000 cm^{-1} (C-Cl stretch): It has bands specific to C-Cl bonds (stretch), being the most relevant region for the characterization of chloroparaffin as a flame retardant.

Region 600–800 cm^{-1} : Strong bands which means a high concentration of chlorine (usually 50–70% for flame retardant applications) that generates strong absorbents. The FTIR spectrum of chloroparaffin (CP) before combustion, used as a flame retardant and component in intumescent systems, reflects only the physical mixture of the components, without chemical modifications, its chemical structure based on chlorinated alkane chains. Before being subjected to heat, chloroparaffin is stable and exhibits absorption bands specific to the C-Cl and C-H bonds.

The FTIR spectrum of liquid PVC (polyvinyl chloride) (Fig. 7), before combustion, shows specific characteristics of the PVC polymer matrix: C-Cl, C-H. In the "liquid" state (often plastisol or dispersion), the spectrum is dominated by vibrations of C-Cl bonds, plasticizers, some additives and possible impurities.

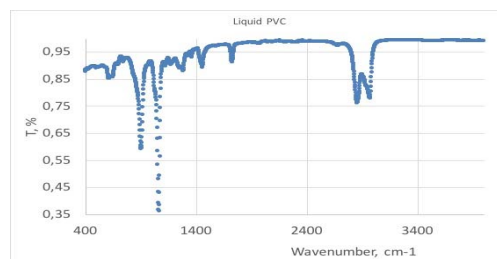


Figure 7. FTIR Spectrum for Liquid PVC

In this state, HCl bands (3000–2700 cm^{-1}) or wide carbon/char bands (far infrared) are not present, combustion bands are absent, they appear only after exposure to heat. The lack of strip at 1600–1635 cm^{-1} (specific to conjugated double bonds) confirms that the polymer has not undergone the dehydrochlorination (degradation) process during mixing

Region 600–700 cm^{-1} (C-Cl area): The stretching vibration of the C-Cl bond that is the main signature of PVC occurs, with minimal distortion that may indicate stereoregular changes or the presence of impurities.

Region 1000–1400 cm^{-1} : The bending vibration CH_2 is highlighted.

Region 2900–3000 cm^{-1} : Exhibits C-H (aliphatic chain) stretching vibrations.

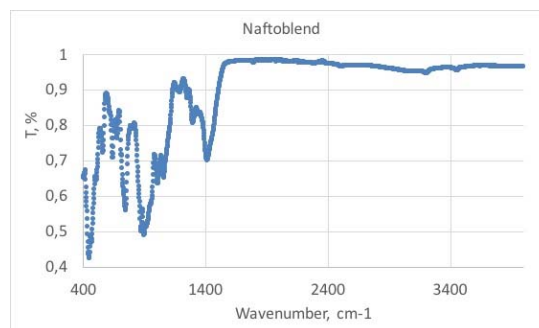


Figure 8. FTIR spectrum for Naftoblend

The FTIR spectrum of Naftoblend ACFR-01 (Fig. 8) (a new intumescent and flame-retardant product, compatible with PVC, polyamides, PP, epoxy resins, etc.), before combustion, highlights the active components intended for the formation of the protective layer (char) and the release of non-flammable gases. The FTIR spectrum is dominated by removals related to phosphorus bonds ($\text{P}=\text{O}$, $\text{P}-\text{O}-\text{P}$), nitrogen ($\text{N}-\text{H}$) and carbonyl ($\text{C}=\text{O}$), which represent the acid-carbon source synergy system. This non-destructive method allows confirmation of the chemical integrity of the flame-retardant additives incorporated into the polymer before they are activated by heat. Naftoblend ACFR-01 is mentioned in the literature as a multifunctional flame-retardant additive, capable of creating protective barriers, and its FTIR spectrum has the above strips superimposed on top of the host polymer spectrum.

Region 1000–1300 cm^{-1} (P=O and P-O-P bonds): These are critical bands, indicative of phosphorus-based flame retardants (e.g. ammonium polyphosphate), essential for dehydration and char formation. Intense bands are observed at $\sim 1200 \text{ cm}^{-1}$ (P=O) and at 1000–1100 cm^{-1} (P-O-P bonds).

Region 1600–1700 cm^{-1} (P-OH bonds): Specific bands for P-OH appear before combustion, showing the presence of the acid source.

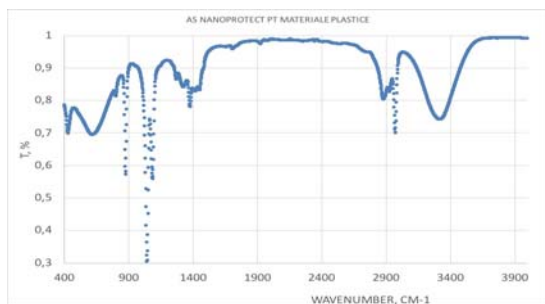


Figure 9. FTIR spectrum for Nanoprotect

The FTIR analysis of this NANO-PROTECT[®] (flame retardant and intumescent) (Fig. 9) fire protection system before combustion indicates the chemical structure of the polymers, blowing agents and nano-additives present in the coating. In the unburned state, the FTIR spectrum highlights the chemical bonds that will react during the fire. Before combustion, the spectrum is characterized by a large number of strips, as all components are present in active form. The purpose of this "cold" layer is to act as a physical barrier (thanks to nanomaterials) and to release chemical compounds (phosphoric acid, inert gases) at high temperatures. Nano-FTIR can be used to identify the distribution of these nanomaterials at a scale of 10-100 nm, showing whether nanosilica or nano-TiO₂ are uniformly dispersed in the binder.

The main FTIR regions:

Region 1000–1300 cm^{-1} , P=O and P-O-C: The characteristic bands of phosphate bonds (from ammonium polyphosphate - APP), essential for fireproofing, appear intensely.

Region 1500–1700 cm^{-1} , N-H and C=N: Groupings of melamine or other foaming agents are highlighted.

Nanoparticles (Nano Component):

Nano-SiO₂ (Silica): Si-O-Si specific bands appear strongly at about 1050–1100 cm^{-1} .

Nano-TiO₂ (Titanium Dioxide): The metal-oxygen (Ti-O-Ti) bands are in the range of 400–900 cm^{-1} .

Surface bonds (Silanization): Silan bands (Si-C) can be seen in the region of 1200 cm^{-1} .

The FTIR spectrum is a combination of organic binders (C-H, C=O bands) and a mixture of inorganic flame retardant (P=O, P-O-P) with nano-particles (Si-O-Si), compatible to react at temperatures of 120°C-200°C, as follows, in Fig10:

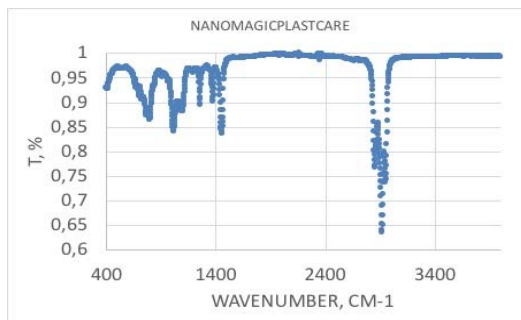


Figure 10. FTIR spectrum for Nanomagiplastcare

Region 1200 - 1500 cm^{-1} (P=O): An intense band, characteristic of the acid source (usually ammonium polyphosphate - APP), is highlighted. This is one of the most important signals for flame retardant properties, showing the presence of phosphorus.

Region 1000 - 1100 cm^{-1} (P-O-P / P-O-C): Vibrations specific to the phosphate structure occur that will form the carbon layer (char)

The presence of nanoparticles (Nano-additives) is evidenced by:

Region $\sim 1000 - 1100 \text{ cm}^{-1}$ (Si-O-Si): The vibration of the silicon-oxygen bond occurs, which possibly overlaps with the phosphate bands, but indicates the presence of nanosilica (nanosilica), used to improve the mechanical strength of the formed char.

Region $< 700 \text{ cm}^{-1}$ (Metal-Oxide): Specific vibrations occur for TiO₂ or complementary nano-structures, which contribute to thermal stability.

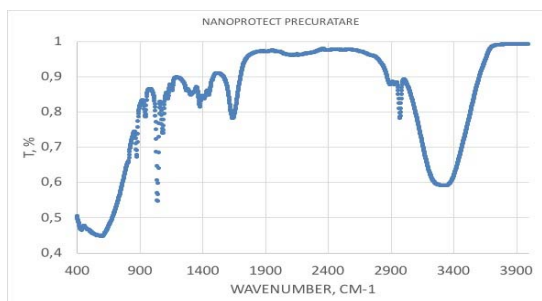


Figure 11. FTIR spectrum for Nanoprotect pre-cleaning

The FTIR spectrum of the Nanoprotect product (Fig. 11), used as an intumescent and flame retardant coating, before combustion, highlights the chemical structure of the polymer (binder) and active flame retardant additives. The purpose of FTIR for Nanoprotect pre-cleaning is to confirm the presence of the key components needed to create the expanded char layer during the fire. Nanoprotect can be considered to be a standard intumescent system, containing an acid source (e.g. Ammonium Polyphosphate - APP), a carbonizing agent (e.g. Pentaerythritol - PER) and a foaming agent (e.g. Melamine - MEL). The pre-combustion FTIR spectrum for Nanoprotect flame retardant/intumescent is characterized by a spectrum rich in groups P=O, P-O-P, N-H and C-H, demonstrating a formulation with

phosphoric acid, polyol and amines (nano inorganic additives).

Region 3100 cm^{-1} , $1500\text{--}1700\text{ cm}^{-1}$ and 900 cm^{-1} (N-H and C=N (Foaming agent): Strips indicating the presence of melamine or its derivatives, which help foaming by releasing inert gases (NH_3).

Region 1800 cm^{-1} – Organic matrix (Binder): Possibly ester, urethane or acrylic strips.

Region 1000 cm^{-1} ($500\text{--}700\text{ cm}^{-1}$), TiO_2 (Titanium Nanoparticles): A wide peak below 1000 cm^{-1} is observed indicating titanium oxide used as a synergistic agent to improve the strength of char.

Region $1000\text{--}1100\text{ cm}^{-1}$, SiO_2 (Nanosilica): Si-O-Si stretch strips that can overlap P-O-P strips, improving thermal stability.

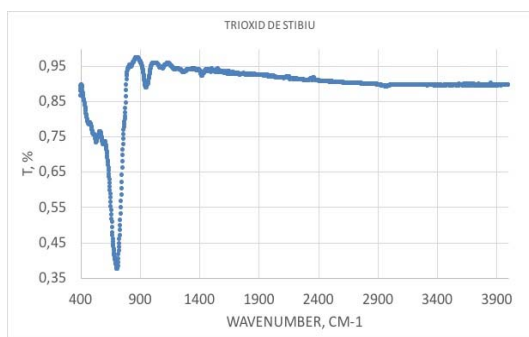


Figure 12. FTIR spectrum of stibium trioxid

In intumescent systems (e.g., combined with ammonium polyphosphate - APP), stibium trioxide is added as synergistic. Before being exposed to the flame, the FTIR

spectrum simply shows the physical mixture of the components, without major structural changes. Its role becomes active only by thermal decomposition, reacting with halogens (if any) to form stibium halides (SbCl_3) in the gas phase. The FTIR (Sb_2O_3) spectrum before combustion (Fig. 12), in the context of flame retardants and intumescent materials, shows characteristic absorption bands in the mid-infrared region ($400\text{--}1000\text{ cm}^{-1}$), corresponding to the vibrations of the Sb-O inorganic bonds.

Sb-O Vibrations (Stretching): The main absorption bands occur at low wavenumbers, identified as Sb–O stretching frequencies, bridge functional groups (O-Sb-O), and symmetrical/asymmetrical vibrations.

Form/lattice tape: The $400\text{--}600\text{ cm}^{-1}$ region shows a widening of the stripes, characteristic of inorganic oxide particles.

4. CONCLUSIONS

This paper has provided a comprehensive and comparative FTIR spectra before combustion, for the flame retardant and intumescent materials, substances. In all cases, a series of absorption bands characteristic of their non-activated organic and inorganic components, which form the protective matrix, leads to the conclusion that they are functional for the intended purpose.

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