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Desarrollo de películas de perovskita con propiedades fotoluminiscentes para aplicaciones en la construcción

Development of perovskite films with photoluminescent properties for building applications

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Resumen-- Materiales energéticamente eficientes como las perovskitas han atraído la atención por sus características, bajos costes de fabricación y variedad de métodos de producción. Estos materiales pueden utilizarse en distintas aplicaciones para promover el desarrollo de ciudades inteligentes. Trabajos anteriores informan de la inestabilidad de las perovskitas expuestas al aire, la humedad y la temperatura, por lo que actualmente se siguen estudiando estructuras con materiales sostenibles que proporcionen una mayor durabilidad. En base a esto, la presente investigación explora la síntesis de un recubrimiento de perovskita en matriz de biopolímero para promover el uso de materiales amigables con el medio ambiente en la construcción. Las películas propuestas se sintetizaron a partir de polisacáridos, acetato de plomo y bromuro de cesio disueltos en un sistema de dimetilsulfóxido y dimetilformamida en condiciones ambientales. Finalmente, se utilizaron diferentes técnicas para estudiar las propiedades ópticas, morfológicas y estructurales del material confirmando su aplicación como películas homogéneas y fotoluminiscentes con un contenido de ligandos orgánicos para la formación de perovskita.

Palabras clave— Huella de carbono; puerto marítimo; transporte marítimo; objetivos de desarrollo sostenible; cambio climático.

Abstract— Energy-efficient materials such as perovskites have attracted attention due to their characteristics, low manufacturing costs and variety of production methods. These materials can be used in different applications to promote the development of smart cities. Previous work reports instability of perovskites exposed to air, humidity and temperature, so currently continues to study structures with sustainable materials that provide greater durability. Based on this, the present research explores the synthesis of a perovskite coating in biopolymer matrix to promote the use of environmentally friendly materials in construction. The proposed films were synthesized from polysaccharides, lead acetate and cesium bromide dissolved in a dimethyl sulfoxide and dimethylformamide system under ambient conditions. Finally, different techniques were used to study the optical, morphological and structural properties of the material confirming its application as homogeneous and photoluminescent films with a content of organic ligands for the formation of perovskite.

Index Terms— Carbon footprint; seaport; maritime transport; sustainable development goals; climate change.

I. INTRODUCTION

THE materials used in civil construction are highly resistant and long-lasting (Akeed et al., 2022). However, with the development of artificial intelligence, smart cities and buildings, it has been investigated that these materials show a

variety of responses to external stimuli, making them not only have a high resistance, but also need to possess optical, antimicrobial and photocatalytic activity, thermo-optical and photometric behaviour, energy conservation and environmental protection (Chiatti et al., 2021; He et al., 2019; Kumar et al., 2020; Liu et al., 2022).

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Thus, in recent years, photoluminescent materials have been studied, because they have the potential to absorb light energy and emit it as visible electromagnetic radiation, which means that traditional electric lighting systems can be replaced by more sustainable ones (J. Yang et al., 2020; Zeng et al., 2020). This property can complement the lighting of buildings, roads and public parks with the coating of concrete with these materials (Han et al., 2017). This is the case of inorganic perovskite metal halides, which have received importance because they have excellent optical and photoluminescence properties, allowing their application in photonics and optoelectronics (Shamsi et al., 2019). Consequently, halide films are ideal for photoluminescent films and inks because they have a large wavelength tuning range (from green to infrared, 400 - 800 nm), long carrier diffusion lengths and synthesis with easy processes at low cost (G. L. Yang & Zhong, 2016).

Particularly, inorganic cesium-lead perovskites have a thermal stability of up to 550 °C, and it is higher compared to hybrids ($CsPbBr_3$ is stable above 500 °C, $MAPbBr_3$ up to 432 °C). They also succeeded in replacing Group II-VI semiconductors for LEDs, lasers, photodetectors, nonlinear applications, solar cells and spectrochemical probes for ion detection (Ananthakumar et al., 2016), (Vashishtha et al., 2020). In this way they are widely known to be an optimal light emitting source and, in addition, the use of perovskite quantum dots improves color purity, light coefficient and is easy to manufacture (Wei et al., 2019).

However, when synthesized, phase segregation, degradation of the photoluminescence, structural and property stability problems can occur when exposed to environments such as air, humidity and variable temperatures, limiting industrial applications and processing opportunities. Therefore, substances that form luminescent components directly inside a matrix such as polymeric components are studied (Wang et al., 2017). Biopolymers that reduce the use of hydrocarbon-based polymers include starch, it is one of the most abundant organic substances in the world, is formed in grains by layers of polymeric macromolecules: amylose (gelatinization) and amylopectin (viscosity) (Ilyas et al., 2018). They have intramolecular organization with H bonds between OH groups,

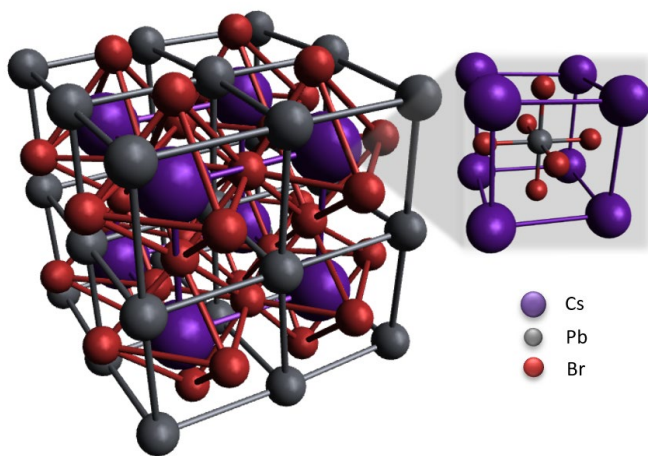


Fig. 1. Perovskite structure $CsPbBr_3$

directly or by water molecules in semi-crystalline structure (dense layers, branching) and amorphous (less organized layers) and is used as a source of energy in tubers like potatoes and plants such as aquatic weeds (Elodea) (Ibrahim et al., 2019; Paveswari & Sithambaranathan, 2011). Use of this could reduce the pollution it causes in lakes, lagoons and rivers. This is the case of Lake Tota in Colombia, which has presented deterioration of water quality and exclusion of less competitive native species due to excessive plants growth caused by the concentration of macronutrients from fertilizers, wastewater discharges, etc. (Castro et al., 2021; O'Hare et al., 2018; Sanabria et al., 2021).

II. METHODS

A. Materials

R.A. Chemicals' 99% lead acetate $Pb(Ac)_2 \cdot H_2O$ and CsBr 99% cesium bromide from Scientific Products. Potato starch was supplied by Brymar SAS. 99.9% anhydrous dimethyl sulfoxide (DMSO) was obtained from Sigma-Aldrich and N,N-dimethylformamide 99.9% (DMF) was acquired from Scientific Products. All materials were used as received without any further purification.

B. Methods

The biopolymer encrusted with perovskite in DMSO and DMF (3:2) is synthesized under environmental conditions. The precursors $Pb(Ac)_2 \cdot H_2O$: CsBr, are prepared with a ratio of 1:3, respectively, for the formation of perovskite in solvents at a concentration of 10% w / w. This mixture is placed on the hot plate with magnetic stirring at 80 °C and 1000 rpm for 30 minutes.

Subsequently, the starch from the aquatic plant (Elodea), extracted by the method described in our previous research (Sanabria et al., 2021) the potato starch are combined in a 1:9 ratio in the quantities of 20% w / w respect to precursors, are added to the container with the previously synthesized perovskite, and there it is left at 80 °C and 1000 rpm for 6 hours.

Once the previous step is completed, 140% of deionized water is added based on the total weight of the mixture. As a result, it precipitates, the crystals are formed and decanted over 15 minutes. The sediment is then applied by coating the glass substrate with the solution and dried onto the hot plate at 100°C for 30 minutes. Finally, 0.2 ml of deionized water is added to the substrate with the perovskite film, that allows the photoluminescence to form and is again left to dry on the plate at 100 °C for 15 minutes.

III. RESULTS

A. Exposure to UV light & Vis

This demonstrated the generation of photoluminescence in the perovskite and biopolymer material. As figure 2 shows, the factors that influence the homogeneity of the film are the deposition method on the substrate, either by using a jet or dripper Spin Coating (Fig 1.a), or by Deep Coating (Fig 1.b), or

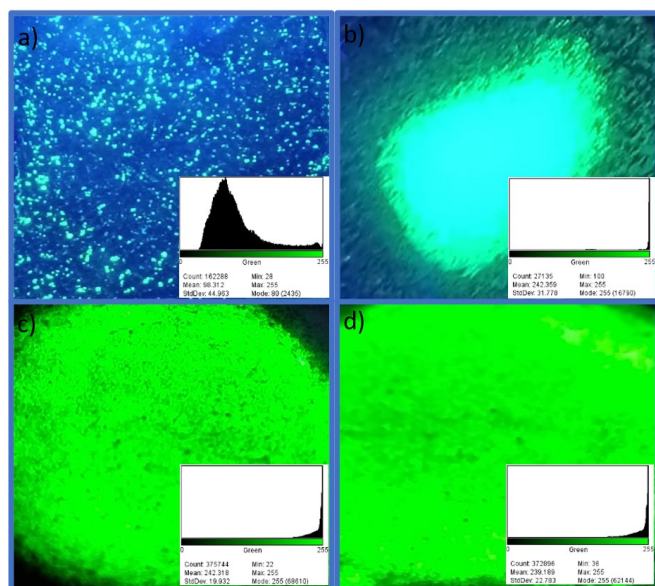


Fig. 2. Progression of photoluminescence

placing the solution on the substrate (Fig 1.c,d), the layers deposited by spin coating, the proportion of precursors and dissolved starches (Konstantakou et al., 2017). It was found that the spin coating jet method allows a more uniform layer of solution than by drip. However, by placing the solution directly on the substrate, when exposed to UV light, a homogeneous photoluminescence is observed (Fig 1. d,c) with percentages of 10% w/w of precursors in the DMSO: DMF solvent system and 20% w/w of starches with respect to precursors.

In this way, the RGB analysis of each sample was carried out, in which, by means of a histogram, the count, average and mode of the pixels that correspond to the characteristic green color of the Bromide perovskite are made, this count is between 0 and 255 pixels. It was found that the sample deposited by Spin Coating had an average of 98 green pixels, by Deep Coating it had a lower pixel count than the others, however the average was 242. Finally, the deposit on substrate found a pixel count higher than the others with an average of 242 (Fig 1.c) and 239 (Fig 1.d), which confirms the uniformity of the perovskite coating on the glass substrate.

The spectrum in Figure 3 shows that peaks of 3472 to 3380 cm^{-1} were found, which initially corresponds to the weak stretching of N-H corresponding to the surfaces of the perovskite $CsPbBr_3$ (Yan et al., 2019). Then there is the OH section, which indicates that the terminations of the hydroxide surface may belong to starch, promoting its use as an organic ligand (G. Li et al., 2016). This typical elongation of the starch OH functional group, with the perovskite composition, shows that the biopolymer interacts with perovskite precursors through hydrogen bonds, thereby improving the film-forming mechanism (Giuri, Masi, et al., 2018; Guo et al., 2020). On the other hand, in this mixture which contains water a pronounced peak can be found because of the OH groups which compose the water, it is possible that for this reason, the samples in which water was added, photoluminescence was produced uniformly, compared with those containing only precursors, starch and solvents.

Moreover, relative to the peaks found in the research of Jeon et al, 2014, in the spectrum, S-O and C-S stretch vibrations of DMSO can be highlighted, these bonds are coordinated with lead at 1000 to 750 cm^{-1} . Finally, stretching N-H is seen in the 1500 cm^{-1} and C-H stretching in the interval from 2996 to 2912 cm^{-1} , indicating the presence of ligands on the perovskite (Jeon et al., 2014; Xiang et al., 2021).

B. Fourier transform infrared spectroscopy (FTIR)

The spectrum in Figure 3 shows that peaks of 3472 to 3380 cm^{-1} were found, which initially corresponds to the weak stretching of N-H corresponding to the surfaces of the perovskite $CsPbBr_3$ (Yan et al., 2019). Then there is the OH section, which indicates that the terminations of the hydroxide surface may belong to starch, promoting its use as an organic ligand (G. Li et al., 2016). This typical elongation of the starch OH functional group, with the perovskite composition, shows that the biopolymer interacts with perovskite precursors through hydrogen bonds, thereby improving the film-forming mechanism (Giuri, Masi, et al., 2018; Guo et al., 2020). On the other hand, in this mixture which contains water a pronounced

TABLE I
PEAKS FOUND IN THE FT-IR

Wavenumber (cm^{-1})	Functional group	Description
3472-3380	O-H voltage vibration and N-H	Weak stretches of N-H are attributed to the surfaces of $CsPbBr_3$
3300		
2996- 2912	C-H voltage vibration	Bands associated with hydrogen bonds of polymerized starch
1662- 1609	C=C voltage vibration	Functional groups corresponding to ligands
1662- 1630	C=O voltage vibration	
1500	N-H deformation	Deformations corresponding to the inclusion of solvent molecules
1000-750	S-O	DMSO sulfonates coordinated with lead
900-690	C-H out of the bending plane	Important links for passivation growth of perovskite crystals (Chen et al., 2019)

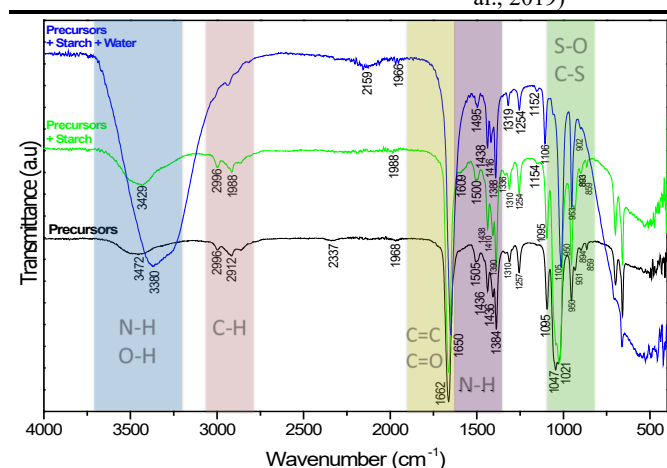


Fig. 3. FTIR a) Precursors in the solvent system, b) Precursors incorporated with starch, c) Precursor mixture, starch and water.

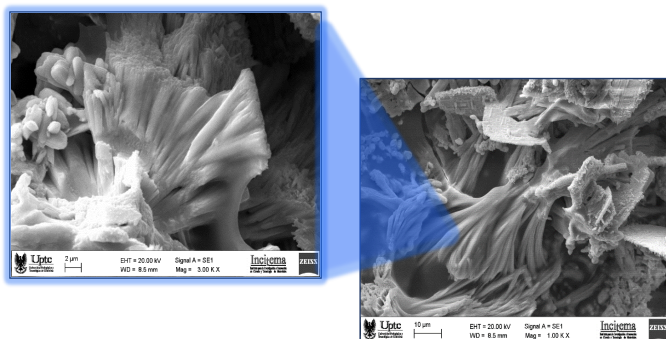


Fig. 4. Scanning electron microscopies at 3000x and 1000x

peak can be found because of the OH groups which compose the water, it is possible that for this reason, the samples in which water was added, photoluminescence was produced uniformly, compared with those containing only precursors, starch and solvents.

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C. Scanning electron microscopy (SEM)

Perovskite particles of different sizes and shapes were observed, found in sticks with an estimated average of $2.45\text{ }\mu\text{m}$, which can be characteristic of perovskites dissolved in DMSO. In addition, this kind of vertical wire structures towards the substrate allows quick separation of holes and electrons, increasing the intensity of light absorption (X. Li et al., 2018). On the other hand, they were also found in cubes of around $10\text{ }\mu\text{m}$ on each side, which may be influenced by DMF (Adhyaksa et al., 2016).

These structures are found in the polymerized starch film, which covers the perovskites, proving the influence of the polysaccharide on crystallization (Giuri, Masi, et al., 2018). However, the shapes and their distribution are not uniform, which means that, although the starch hydroxyl groups coordinate with lead and solvents, what was confirmed in the FTIR spectra (Figure 4), crystalline growth is uncontrolled, and this can happen because the concentration of the organic ligand is not enough (Giuri, Colella, et al., 2018).

Additionally, Energy Dispersive X-ray Spectroscopy (EDS) is required for this type of substrate coating, because in this type of nano and microscopic scales, pinholes may be present. By performing the complete tracking or mapping of the substrate, the uniform distribution of the elements can be confirmed. This is how, through the scanning of SEM-EDS are corroborated in addition to the elements that compose the film, the homogeneity of these elements on the substrate.

This mapping allowed us to link oxygen and carbon to starch, and bromide, cesium and lead that belong to perovskite, (Table 2). In this way, the dissolution of potato and Elodea polysaccharides is evidenced, showing the formation of hydrogen bonds between the molecules of dimethyl sulfoxide

TABLE I
COMPOSITION OF BIOPOLYMER PEROVSKITE FILM (SEM-EDS)

Element	Weight percentage (%)	Atomic percentage (%)
C	16.42	50.34
O	10.86	25.01
Br	18.34	8.46
Cs	24.47	6.78
Pb	26.46	4.70
Total	100.00	

with the first layer of starch which is amylopectin, allowing the release of amylose, which causes the swelling of the granules and finally the gelatinization. Secondly, the structure of the perovskite can be corroborated, finding that the atomic percentage of the bromide represents the highest proportion of atoms in relation to the total number of atoms present in the composite perovskite.

IV. CONCLUSIONS

A method of synthesis of inorganic perovskites CsPbBr_3 from Lead Acetate and Cesium Bromide was developed under environmental conditions using starches as an organic ligand for their formation.

Starch- DMSO film: in the dissolving of polysaccharides (Elodea and potato), there is the formation of H-bonds between DMSO molecules with the first layer of starch that is amylopectin, allowing the release of amylose, which causes the granules to swell and eventually gelatinization.

By incorporating starch with precursors and solvents, the mixture transforms the characteristic green color of bromine halide perovskites, and by IR spectra, the coordination of the self-bonds that form the perovskite is confirmed.

This research opens the possibility of new technologies in which materials abundant in nature are incorporated as an alternative to hydrocarbon-based products, the transition to developing energy efficient materials for their application as concrete coating for smart buildings, implementing simpler processes for obtaining and using accessible reagents such as lead acetate and DMSO.

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