



**International Journal of Biology, Pharmacy
and Allied Sciences (IJBPAS)**

'A Bridge Between Laboratory and Reader'

www.jibpas.com

MORPHOLOGICAL INSIGHTS INTO BIODEGRADABLE POLYMERIC FILMS: A COMPARATIVE STUDY OF PLA, CELLULOSE, AND STARCH USING SEM AND X-RAY DIFFRACTION

AHUJA S*, KHANT HB AND RAULJI HJ

Department of Chemistry, Parul Institute of Applied Science, Parul University, Waghodiya,
Gujarat, India

*Corresponding Author: Dr. Sonam Ahuja: E Mail: sonam.ahuja82106@paruluniversity.ac.in

Received 4th Nov. 2023; Revised 5th Dec. 2023; Accepted 2nd May 2024; Available online 1st Feb. 2025

<https://doi.org/10.31032/IJBPAS/2025/14.2.8682>

ABSTRACT

Using a variety of analytical techniques, this research report presents a thorough characterization investigation on biodegradable polymeric films based on polylactic acid (PLA), cellulose, and starch. The films' crystalline structure and molecular arrangement were investigated using X-ray diffraction (XRD), which provided information on their mechanical and thermal characteristics. The surface morphology could be seen in great detail thanks to scanning electron microscopy (SEM), which also helped to better understand the microstructure of the films. FTIR, or Fourier Transform Infrared Spectroscopy, was used to look at the chemical makeup and bonding relationships in the films. The investigation explores the molecular vibrational modes, clarifying the existence of functional groups and their influence on the general characteristics of the films. Furthermore, UV-Vis analysis was carried out to assess the optical qualities, including transparency and absorption qualities, providing insight into the films' possible uses in packaging and other optical devices. The combined use of these cutting-edge characterization methods offers a comprehensive understanding of the structural, morphological, chemical, and optical characteristics of PLA, cellulose, and starch-based polymeric films. A thorough grasp of these biodegradable materials' qualities is essential for customizing them for a range of uses, especially when it comes to ecologically friendly substitutes for typical plastics and sustainable packaging.

Keywords: cellulose, PLA, packaging

1.0 INTRODUCTION:

Cellulose, starch, and PLA (Polylactic Acid) are all biodegradable polymers with various applications in the field of materials science and biotechnology [1]. Cellulose is a natural polymer found in the cell walls of plants. It consists of repeating glucose units and is known for its remarkable strength and biodegradability. Recent research has focused on extracting cellulose from various sources, including agricultural waste, for use in eco-friendly materials and packaging. For the latest developments, I recommend checking scientific journals and databases like PubMed for articles published after my knowledge cutoff date in September 2021 [2]. Starch is another natural polymer made up of glucose units. It's abundant in sources like corn and potatoes. Starch-based materials have gained attention for their biodegradability and potential use in bioplastics. To find the latest research on starch-based materials, you can search academic databases and journals [3]. PLA (Polylactic Acid) is a synthetic biodegradable polymer derived from renewable resources like cornstarch or sugarcane. PLA has become popular in 3D printing, packaging, and biomedical applications. To get the most up-to-date information on PLA and its applications, look for recent articles in materials science journals

and conference proceedings. Remember that the field of biodegradable polymers is rapidly evolving, so it's essential to access the latest research articles and reviews to stay informed about the current state of these materials. Biodegradable plastic is different from a compostable one in this way. In fact, during the composting stages, compostable plastic is degraded by biological processes along with other known organic materials to produce biomass that releases carbon dioxide, water, and an organic soil amendment rich in humic substances. This happens in an environment free of any toxic residues and where the microorganisms are present at a constant rate but cannot be seen.

Because of this, all biodegradable polymers are also compostable [4]. Natural polymer : Banana leaf the scientific name of banana leaf is *Musa sapientum* leaf which is obtained from banana plant.

In tropical and subtropical nations, the banana is one of the most widely available and consumed fruits with its high concentrations of minerals, vitamins, carbs, flavonoids, and phenolic compounds. Banana leaves can be used as a source of raw materials for the green technology industry because they are widely accessible and readily available in big quantities. Banana leaf extraction typically

refers to the process of obtaining useful components or materials from banana leaves. This can involve various methods, including (1) Fiber Extraction: Banana leaves can be processed to extract natural fibers that are used for making traditional crafts, textiles, and even paper, (2) Medicinal Use: In some traditional medicine practices, banana leaves are extracted to obtain compounds believed to have medicinal properties [5].

2.1 MATERIAL & METHOD:

2.2 Material:

PLA (Polylactic Acid) used in this study was sourced from Bankabio limited Hyderabad, India. The Cellulose purchased in this study was obtained from Tarapur MIDC Boisar, Palghar, Maharashtra, India. Starch was brought from Pallav chemicals and solvents Pvt Ltd near MIDC Tarapur Boisar, Maharashtra, India.

2.3 Method:

First, we have taken cellulose, starch and PLA polymer. Four different ratios were exposed mainly 60:30:10,70:20:10,80:10:10 and 100:0:0. PLA generally not soluble in water, so we have to dissolve PLA in 50 ml of chloroform. After taking 60 grams of PLA, placing it in a conical flask at 25 degrees Celsius, and Stirring was initiated to facilitate the process, and as PLA dissolved in chloroform, the solution began to take form.

Subsequently, 30% cellulose was introduced into the solution and carefully stirred until a homogenous mixture was achieved. Following this step, 30 ml of starch was added to the solution. The reaction resulted in the formation of a solution with distinct characteristics. This solution was then transferred into a Petri dish, and the dish was placed in an oven maintained at room temperature for a duration of 30 to 60 minutes and like that we have made all film. To create a natural drug, we embarked on the process by selecting a key ingredient, banana leaves (kela ka paan). The leaves were carefully collected and placed in an oven for drying to remove moisture content, ensuring the efficacy of the extraction. Once the leaves were adequately dried, they were meticulously crushed to increase the surface area for extraction. In a separate beaker, ethanol was introduced, and the crushed banana leaves were mixed into the solvent. The resultant mixture was subsequently filtered to separate the solid plant material from the liquid extract. This liquid extract represented the essence of the natural drug, containing the potentially valuable compounds from the banana leaves. To fabricate a simple film for the delivery of our natural drug, we adopted a composition of 80:10:10 ratio of a solution. The natural drug was initially obtained through a meticulous

extraction process, with the resulting liquid containing 3 ml of the drug solution. These components were thoroughly combined to form a homogenous mixture.

3.0 Chemical Analysis:

3.1 Swelling Study:

The study examined the swelling of composite films using physiological fluid (PF). This fluid was made by dissolving 8.307 g of sodium chloride (NaCl) and 0.367 g of calcium chloride (CaCl₂) in one liter of distilled water. After that, they were mixed to produce the PF solution. Discrete weighted little portions of every ratio film were now cut. The films were weighed after being soaked in the PF solution and periodically removed to be dried on filter paper. This process was carried out several times and once every 24 hours.

3.2 Dynamic moisture adsorption studies:

In the course of this experiment, a fascinating relationship between the solubility of KNO₃ (Potassium Nitrate) and temperature was explored. The procedure began with the preparation of a solution by adding KNO₃ to 100 ml of water, followed by crushing the substance into small fragments. The solution was then transferred into a plastic container, while the crushed KNO₃ pieces were placed in a petri dish. This petri dish was sealed within the plastic container, creating a controlled environment. Over the course of

the experiment, temperature readings were diligently recorded at 15-minute intervals.

3.3 SEM (Scanning Electron Microscopy):

Scanning electron microscope (SEM) is one of the most widely used instrumental methods for the examination and analysis of micro- and nanoparticle imaging characterization of solid objects. One of the reasons that SEM is preferred for particle size analysis is due to its resolution of 10 nm, that is, 100 Å [10]. To ascertain the morphological characteristics of the composite film, PNP Analytical Solutions, Vadodara, carried out a scanning electron microscopy investigation.

3.4 XRD:

A widely used XRD analysis technique is the identification of materials using their diffraction pattern. XRD provides information on how internal stresses and flaws cause the real structure to differ from the ideal one in addition to phase identification. Whereas crystals are ordered arrangements of atoms, X-rays are electromagnetic energy waves. Crystal atoms scatter when incident X-rays collide with their electrons. Elastic scattering is the process where the electron acts as the scatterer. A regular array of spherical waves is produced by a regular array of scatterers. In most directions, these waves cancel each other out due to destructive interference. Furthermore, they add constructively in a few

specific ways, according to Bragg's law [11]. XRD analysis performed at PNP analytical solution at Vadodara.

4.0 RESULT AND DISCUSSIONS:

4.1 Swelling Study:

The definition of swelling is a rise in the volume of a solid or gel caused by the absorption of a liquid or gas. The swelling investigation was conducted using the methodology outlined in this paper's Characterization section (**Table 1 and Figure 1**). The formula was used to get the Ratio of Swelling, or SR. $SR = (M_t - M_0) / M_0$ g/g where M_0 = mass at initial and M_t = mass at interval t. Here Ratio of Film in PLA: CELLULOSE: STARCH respectively.

4.2 Dynamic Moisture Adsorption Studies:

It is obvious from looking at the graph that there is an upward tendency. In this study, sol-gel and atmospheric drying technologies were used to create fiber felt/silica aerogel composites with extremely low moisture adsorption rates. Particularly in humid regions, the produced fiber felt/silica aerogel composites with ultra-low moisture absorption rate can be widely used as building insulation **Table 2**.

4.3 UV-Visible:

UV absorption is a commonly used method in analytical chemistry to determine the presence and concentration of various compounds in a

solution. In this case, the range of 410 to 500 nanometres is likely indicative of the wavelengths at which the banana leaf extract absorbs UV light and it is also a baseline water [12] **Figure 2**.

4.4 FTIR SPECTRUM:

Poly (lactic acid) (PLA) often shows many distinctive peaks at different wavelengths, including both wide and sharp peaks, in its Fourier-Transform Infrared (FTIR) spectrum. The significant peak in PLA's FTIR spectrum that is linked to hydrogen bonding between polymer chains is one of its most noticeable characteristics. We can see PLA spectrum in below Figure (). In this Figure we can observe a sharp peak nearly at 1747 cm^{-1} which is shows that C=O carbonyl group is present in PLA. The stretching vibrations of the methyl (CH_3) groups found in PLA seen between 2895 and 2995 cm^{-1} . Usually, these peaks are sharp. At 1078.61 and 1180 cm^{-1} , peaks linked to the stretching vibrations of the carbon-oxygen-carbon (C-O-C) and oxygen-carbon-oxygen (O-C-O) links in PLA can be recognized. Some of these peaks may be rather pointed. Now we can see in PLA spectrum -OH group is presented in between 3000 to 3500 cm^{-1} with broad peak. The PLA exhibits many C-H bending and rocking modes, which can give rise to peaks with varying shapes and sharpness at 1449 and

1356 cm⁻¹ [13]. FTIR spectrum shown in **Figure 3**.

4.5 THERMOGRAVIMETRY

ANALYSIS:

The practical usefulness of a linearizing approach like the one described here in classifying and identifying the different deviations that are always present in experimental data can only be established with significantly more experience. A basic decomposition strategy may be relatively effectively hidden when numerous nonrandom experimental mistakes of the kind outlined contribute large deviations to the data. However, the outcomes with a small number of systems have been quite promising the technique has at the very least shown to be helpful in differentiating between random and intentional variances in thermograms [14].

Figure 4.

4.6 SEM ANALYSIS:

In scanning electron microscopy, an electron beam is used to scan a material, creating an

observable magnified image. The electron micrographs of the pectin/chitosan composite film's surface are displayed in Figure --. It was discovered that the particles in the composite film had sizes between 300 and 600 nm. It is clear that there are no noticeable agglomerations or rough areas in the surface morphology [15]. Here the result of SEM analysis in **Figure 5**.

4.7 X-RAY DIFFRACTION ANALYSIS:

In material science, X-ray diffraction analysis is a technique used to ascertain a material's crystallographic structure. By subjecting the sample to X-rays, this approach analyzes the rays' intensity and dispersion as they exit the material. The XRD spectrum of a composite Pectin/PVA film is shown in **Figure 3**. A hump in the spectra at 19.5 ($2\theta=5.0485$) was observed using an X-ray diffractometer, which is consistent with the amorphous nature of the film [16]. XRD graph is shown in **Figure 6**.

Table: 1 Swelling Study

Sr. No.	Time	80:10:10	70:20:10	60:30:10	PLA
1	30 min	5.15 gm	4.21 gm	4.66 gm	5.93 gm
2	60 min	7.63 gm	6.08 gm	6.57 gm	7.25 gm
3	90 min	9.26 gm	7.60 gm	8.38 gm	8.31 gm
4	120 min	10.21 gm	8.52 gm	9.28 gm	9.12 gm
5	150 min	11.63 gm	9.65 gm	10.38 gm	9.93 gm
6	180 min	12.19 gm	10.50 gm	11.52 gm	10.81 gm

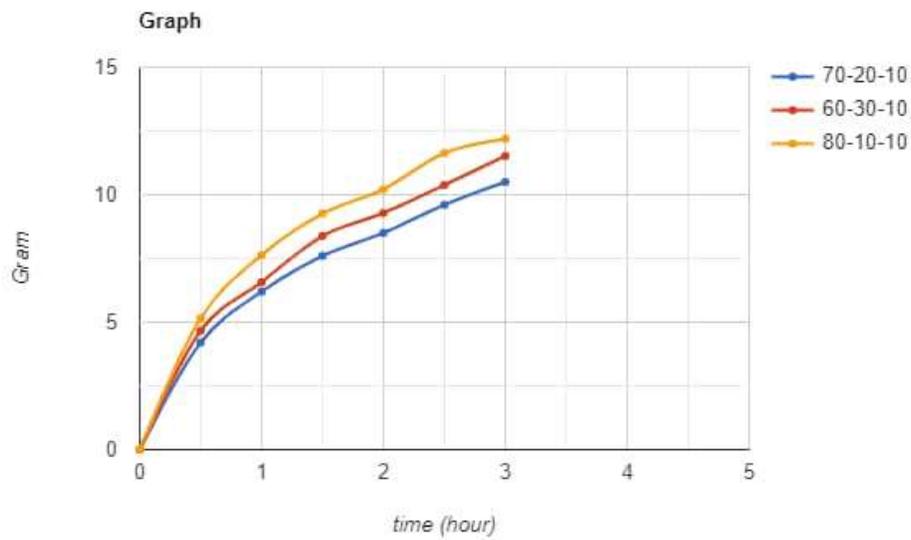


Figure 1: Graph of Swelling Study

Table 2: Dynamic Moisture Adsorption Studies

Sr. No.	Time	80:10:10	70:20:10	60:30:10	PLA
1	0 min	0.7 gm	0.10 gm	0.6 gm	0.9 gm
2	15 min	0.9 gm	0.11 gm	0.8 gm	0.10 gm
3	30 min	0.11 gm	0.12 gm	0.9 gm	0.11 gm
4	45 min	0.12 gm	0.14 gm	0.10 gm	0.13 gm
5	60 min	0.14 gm	0.15 gm	0.12 gm	0.14 gm

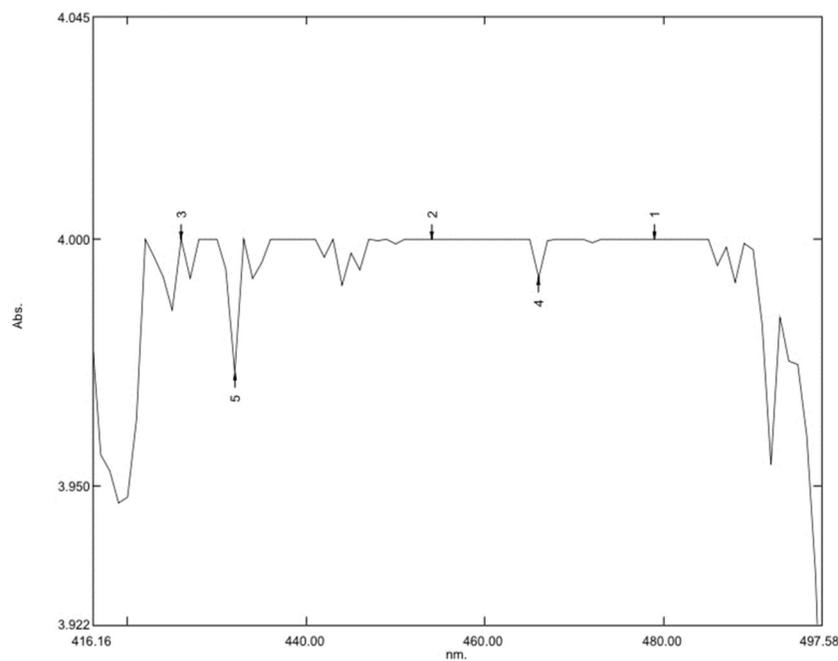


Figure 2: Ultra violet Absorption Graph

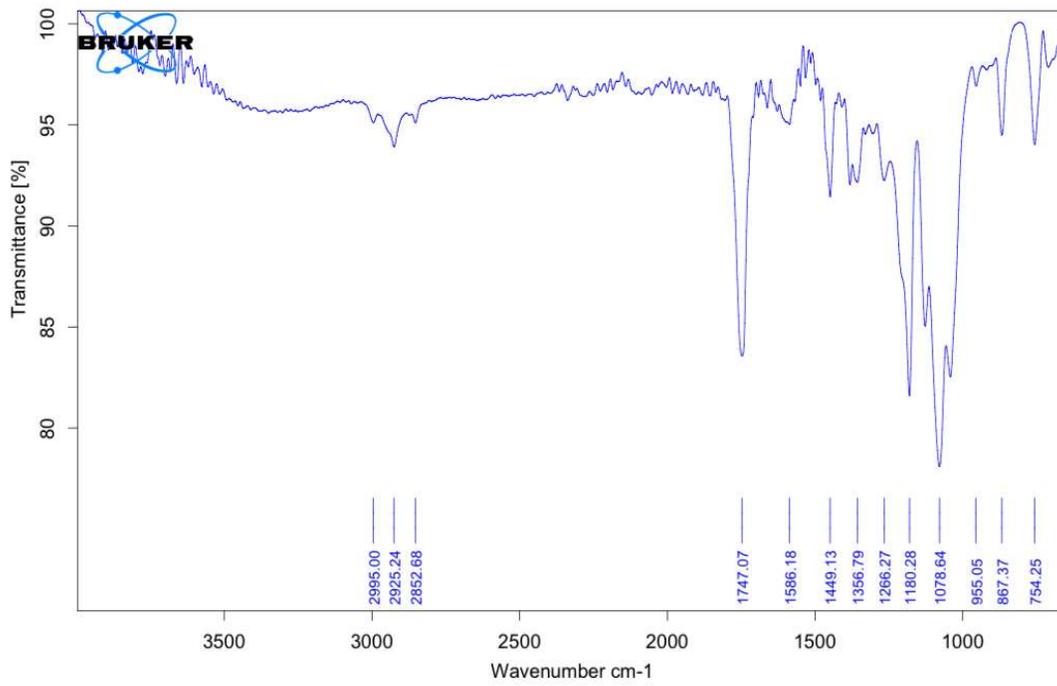


Figure 3: FTIR Spectrum of Poly Lactic Acid

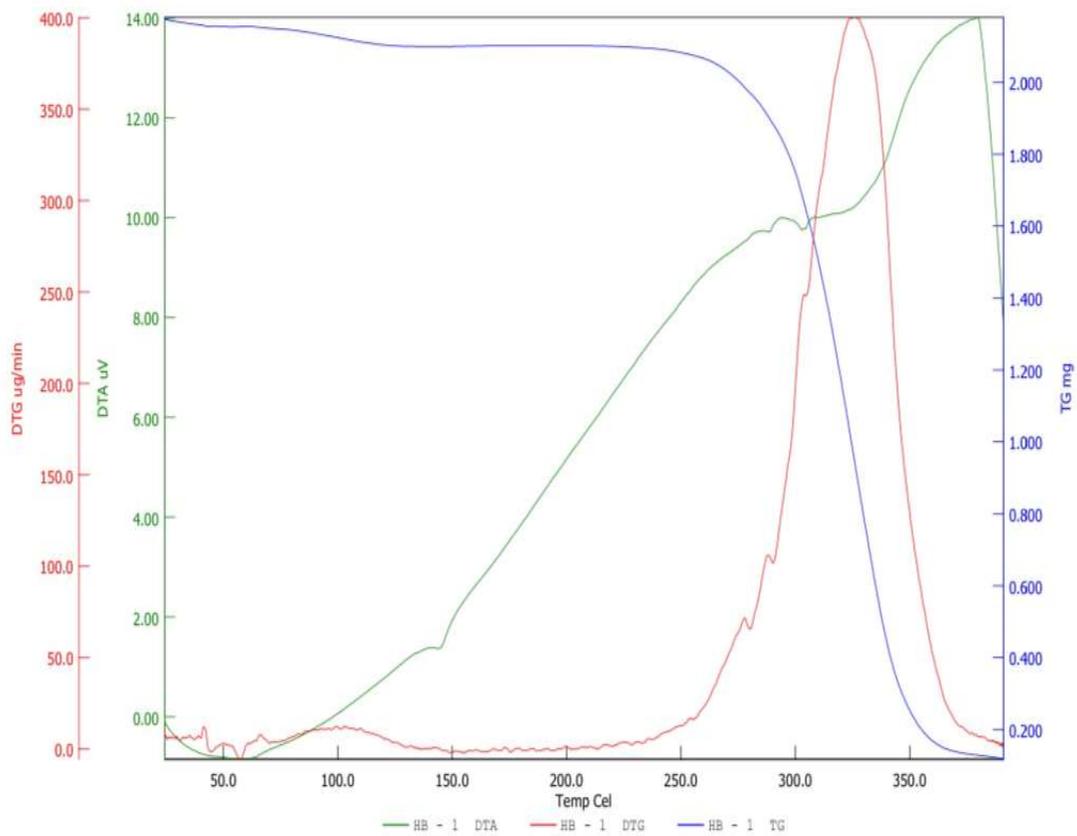


Figure 4: Graph of Thermogravimetry Analysis

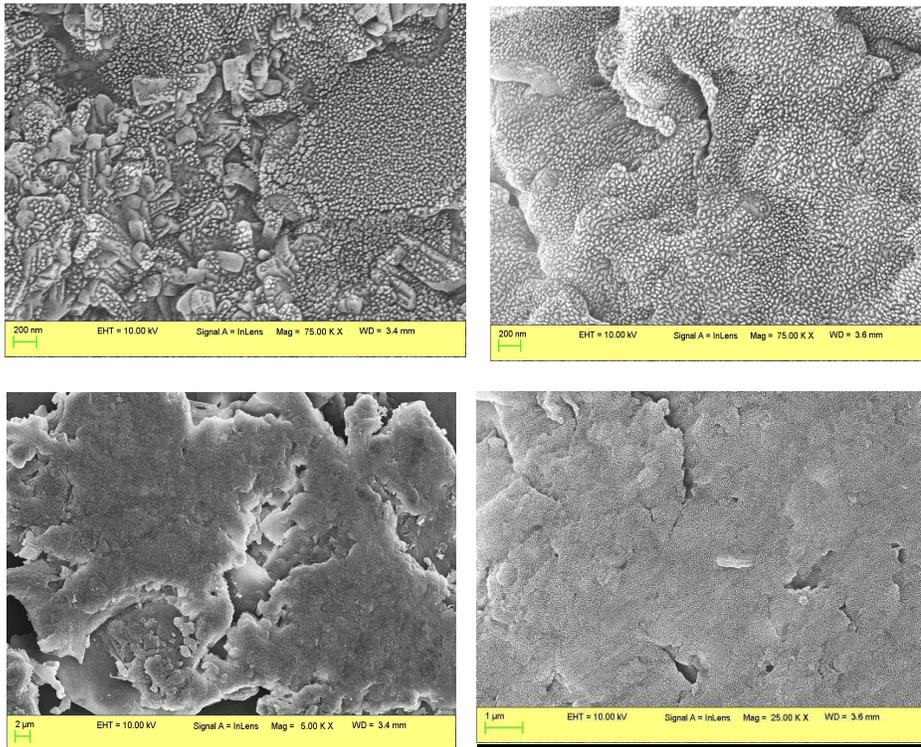


Figure 5: SEM Analysis

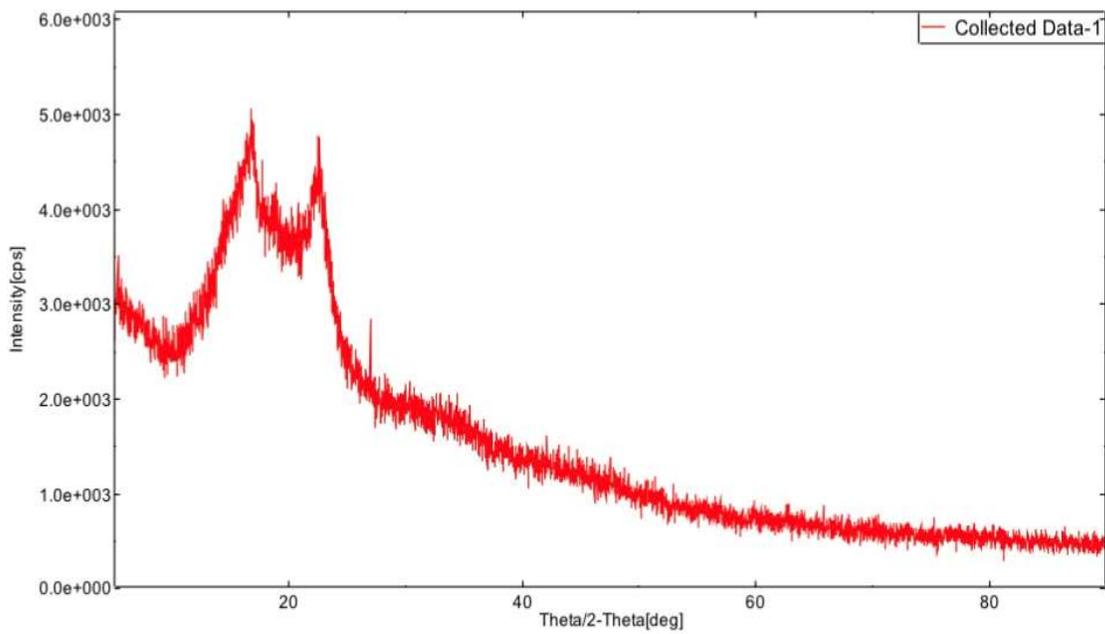


Figure 6: Graph of X-ray Diffraction

CONCLUSION:

To sum up, the PLA, cellulose, and starch-based biodegradable polymeric films display unique properties that are essential for a variety of uses. Variations in swelling studies' capacity to absorb water highlight their potential for use in controlled release systems. The crystalline nature of cellulose is highlighted by X-ray diffraction, which affects material strength. Unique surface morphologies are highlighted by scanning electron microscopy, which helps evaluate the functionality of films. The chemical composition and intermolecular interactions are verified by FTIR analysis. The optical characteristics are demonstrated for possible sensor applications using UV-vis analysis. All things considered, the thorough characterization clarifies the complex nature of these biodegradable films and offers insightful information for biological, industrial, and ecologically friendly packaging applications.

Acknowledgement:

I would like to thank to Parul University for providing FTIR facilities and also thank to the PNP analytical solution, Vadodara for the XRD and SEM analysis.

REFERENCES:

[1] Masmoudi F, Bessadok A, Dammak M, Jaziri M, Ammar E. Biodegradable

packaging materials conception based on starch and polylactic acid (PLA) reinforced with cellulose. *Environmental Science and Pollution Research*. 2016 Oct;23:20904-14.

[2] Moon RJ, Schueneman GT, Simonsen J. Overview of cellulose nanomaterials, their capabilities and applications. *Jom*. 2016 Sep;68:2383-94.

[3] Pfister B, Zeeman SC. Formation of starch in plant cells. *Cellular and Molecular Life Sciences*. 2016 Jul;73:2781-807.

[4] Murariu M, Dubois P. PLA composites: From production to properties. *Advanced drug delivery reviews*. 2016 Dec 15;107:17-46.

[5] Yingyuen P, Sukrong S, Phisalaphong M. Isolation, separation and purification of rutin from Banana leaves (*Musa balbisiana*). *Industrial Crops and Products*. 2020 Jul 1;149:112307.

[6] Passos ML, Saraiva ML. Detection in UV-visible spectrophotometry: Detectors, detection systems, and detection strategies. *Measurement*. 2019 Mar 1;135:896-

[7] Singh VK, Rai PK. Kidney stone analysis techniques and the role of

- major and trace elements on their pathogenesis: a review. *Biophysical reviews*. 2014 Dec;6(3-4):291-310.
- [8] Pasquini C. Near infrared spectroscopy: A mature analytical technique with new perspectives—A review. *Analytica chimica acta*. 2018 Oct 5;1026:8-36.
- [9] Pasquini C. Near infrared spectroscopy: A mature analytical technique with new perspectives—A review. *Analytica chimica acta*. 2018 Oct 5;1026:8-36
- [10] O'Connell RC, Dodd TM, Clingerman SM, Fluharty KL, Coyle J, Stueckle TA, Porter DW, Bowers L, Stefaniak AB, Knepp AK, Derk R. Developing a Solution for Nasal and Olfactory Transport of Nanomaterials. *Toxicologic pathology*. 2022 Apr;50(3):329-43
- [11] Vashistha P, Oinam Y, Kim HK, Pyo S. Effect of thermo-mechanical activation of waste concrete powder (WCP) on the characteristics of cement mixtures. *Construction and Building Materials*. 2023 Jan 2;362:129713.
- [12] Mishra G, Praharaj P, Pandey S, Parida S. Biodegradable layered double hydroxide based polymeric films for sustainable food packaging applications. *Applied Clay Science*. 2023 Aug 1;240:106978.
- [13] Taib NA, Rahman MR, Huda D, Kuok KK, Hamdan S, Bakri MK, Julaihi MR, Khan A. A review on poly lactic acid (PLA) as a biodegradable polymer. *Polymer Bulletin*. 2023 Feb;80(2):1179-213.
- [14] Oliviero M, Lamberti E, Cafiero L, Pace B, Cefola M, Gorrasi G, Sambandam A, Sorrentino A. Biodegradable cellulose acetate/layered double-hydroxide composite film for active packaging of fresh food. *Materials Chemistry and Physics*. 2023 Dec 1;310:128469.
- [15] Yanat M, Muthurajan M, Strubel M, Grolle K, Schroën K. Polylactic acid films reinforced with chitin nanocrystals: Biodegradation and migration behavior. *Food Packaging and Shelf Life*. 2023 Dec 1;40:101217.
- [16] Khalili H, Bahloul A, Ablouh EH, Shehaqui H, Kassab Z, Hassani FZ, El Achaby M. Starch biocomposites based on cellulose microfibrils and nanocrystals extracted from alfa fibers (*Stipa tenacissima*). *International Journal of Biological Macromolecules*. 2023 Jan 31;226:345-56.