International Journal of Innovative Research in Engineering and Management (IJIREM)
ISSN (Online): 2350-0557, Volume 12, Issue 4, August 2025
DOI: https://doi.org/10.55524/ijirem.2025.12.4.10
Article ID IJIR3120, Pages 55-62
www.ijirem.org

Green Synthesis and Characterization of Novel Biodegradable Film Based on Renewable Crosslinking Approach

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Received: 2 August 2025 Revised: 16 August 2025 Accepted: 31 August 2025

ABSTRACT- Petroleum-based synthetic polymers are now the best choice for food packaging because they are easy to work with and cheap to make. But these polymers don't break down naturally, which makes it hard to deal with trash after it's been thrown away. A deep need is felt about biodegradable material. Because it is abundant, renewable, and biodegradable, starch could be an ideal material for making biodegradable polymers. PVA is water soluble polymer and has excellent properties, such as being able to emulsify adhesive and film forming ability. In the present study, biodegradable film was developed from potato starch and PVA cross-linked with a bio-crosslinker. The native starch has some drawbacks like brittleness, moisture sensitivity, very less tendency to film forming and low mechanical properties. To overcome this problem, starch was cross linked with oxidised sucrose. With several aldehyde groups per molecule, oxidized sucrose is a green cross-linker that forms hemiacetals and acetals to cross-link starch molecules. Film were developed with oxidised sucrose via solution casting. The addition of 3% cross linker exhibited higher tensile and elongation when compared to the one without cross linker. The casted films were further characterized by SEM, FTIR and TGA. The developed films represent high potential alternative to existing non-biodegradable plastics particularly for packaging applications.

KEYWORDS- PVA, Starch, Oxidized Sucrose, FTIR, Biodegradable film

I. INTRODUCTION

Interest in preserving the environment has been rising consistently and quickly in recent years, not only via the use of items created from naturally renewable resources but also through the use of products that decompose into environmentally friendly components. In practically every industrialized nation have leaped up green programs, policies, and practices to reduce the amount of solid polymeric waste that consumers produce annually. Additionally, consumers have shown a desire for goods that are environmentally friendly while still delivering the same outcomes as goods produced of synthetic materials. However, the higher price and inferior qualities of environmentally friendly products compared to those made from synthetic materials can discourage consumers from buying them [1]. Mankind has employed biopolymers, particularly the fibrous ones, since the dawn of civilization. [2]. One of the most common polysaccharides is starch, which is composed of numerous repeating units of the monosaccharide glucose and is connected to other molecules by alpha-(1,4) glycosidic linkages. Amylose and amylopectin are the two polysaccharides that make up starch [3]. Starch films and starch granules are partially solubilized as a result of this. Due to its high moisture sensitivity and low water stability, starch's hydrophilic nature has a negative impact on the production of starch-based products. Secondly, it has poor mechanical qualities, such as low elongation, low strength, and brittleness, due to the amorphous regions in the structure generated by amylose and branching sites of amylopectin. As a result, native starch-sized yarns cannot be woven using high speed techniques. Numerous investigations to address these issues and improve the starch-based products' suitability for industrial uses. In order to address the aforementioned drawbacks of native starch, improve its adaptability, and meet market demand, starch is modified. To create modified starches with various properties for a variety of applications, numerous techniques have been devised. The five categories of starch modification strategies include physical, chemical, grafting, enzymatic, and genetic changes [4]. Usually, physical changes to starch are employed to change its granular structure and increase its solubility in cold water. They don't require any chemical processing, and they're also affordable and easy. Native starch can be physically altered by a number of processes, including heat moisture treatment, freezing, ultra-high-pressure treatment, annealing, retrogradation, and gelatinization [5]. Starch is an example of a polymer that contains hydroxyl groups, and in order to crosslink it, the crosslinking agent needs to be able to react with at least two of these groups in a single polymer molecule [6]. One of the most popular methods to improve the mechanical characteristics and water resilience of starch films is crosslinking. For the crosslinking of starch, this happens when a bifunctional or multifunctional crosslinking agent binds to the both primary or the secondary hydroxyl groups generate intermolecular connections. As a result, crosslinking strengthens the bonds between starch chains and binds the molecules together. PVA is utilized in packaging and as an optical polarizer due to its advantageous features. This polymer is also useful in other fields, such as adhesives, binding and coating, recycling of plastics, and food packaging. Despite being a synthetic polymer made from petroleum, PVA is a form of biodegradable resin. PVA is widely used in a variety of scientific and technological disciplines, including finishing and coating agents, wood, emulsifiers, and leather. It is a great source of organic, surface, textile and fiber materials [7]. In the present study aimed to prepare a biodegradable film based on PVA/Starch crosslinked with oxidized sucrose. The biodegradable film was tested by Tensile properties, Thermal, Surface morphology and biodegradable properties.

II. MATERIAL AND METHOD

A. Materials

Potato tubers (*Solanum tuberosum L.*) were freshly procured from the local vegetable market in Bengaluru, India, and used as the primary raw material for starch extraction. Polyvinyl alcohol (PVA; molecular weight ~85,000–124,000; ≥99% hydrolyzed) and glycerol (≥99.5% purity, analytical grade) were supplied by LOBA Chemie Pvt. Ltd., Pune, India. Sucrose (analytical grade) and sodium periodate ($NaIO_4$, ≥99% purity) were obtained as a generous contribution from Central Drug House Pvt. Ltd. (CDH), Mumbai, India. Distilled water prepared in the laboratory was employed for all extractions, washing, and formulation processes to ensure the absence of ionic contaminants.

B. Experimental Methodology

- Extraction of starch from potato- The potatoes were thoroughly washed with distilled water, peeled, and grated using a stainless-steel grater. The grated mass was homogenized with distilled water in a mortar and pestle to facilitate the release of starch granules. The resultant slurry was filtered through a fine mesh tea strainer to separate fibrous residues. The filtrate was collected in a beaker and allowed to stand undisturbed for 20 min to promote sedimentation of starch. After sedimentation, the supernatant was carefully decanted, and the starch sediment was resuspended in distilled water. The suspension was stirred and subjected to repeated washing–decantation cycles until a clean starch fraction was obtained. The final starch sediment was dried in a hot air oven at 50–60 °C until constant weight was achieved. The dried starch was then ground into fine powder and stored in airtight containers for further use. Residual potato pulp was reprocessed using the same procedure to enhance starch recovery.
- Modification of Sucrose- Sucrose was oxidized via periodate cleavage. For this, 5 g of sucrose was dissolved in 100 mL of distilled water, followed by the addition of 10 g sodium periodate. The reaction mixture was stirred at room temperature for 24 h. Subsequently, 5 g of barium chloride was added, and stirring was continued at 5 °C for 1 h to complete precipitation.
- Preparation of film- For preparation of films, in figure 1 we followed three steps. In first step the required amount of starch 1 g, 2 g, 4 g were added with 100 ml distilled water and was stirring 30 mins at 75°C with the help of magnetic stirrer. In second step for crosslinked films, we added required amount of crosslinker (1%, 2%, 3% dry weight of starch). And in third step required amount of PVA (1.5 g, 2.5 g, 3.5 g) had been melted and added to the starch solution. The plasticizer was adding after 5 minutes, and it was stirred for 30 minutes. The proper mixed solution was pouring in glass petri dish and kept in oven for 12 hours at 50°C. for proper crosslinking we had to put crosslinked solution at 120°C for 10 mins and then 24 hours at 50°C. At last, the film was taken out of the petri dish.

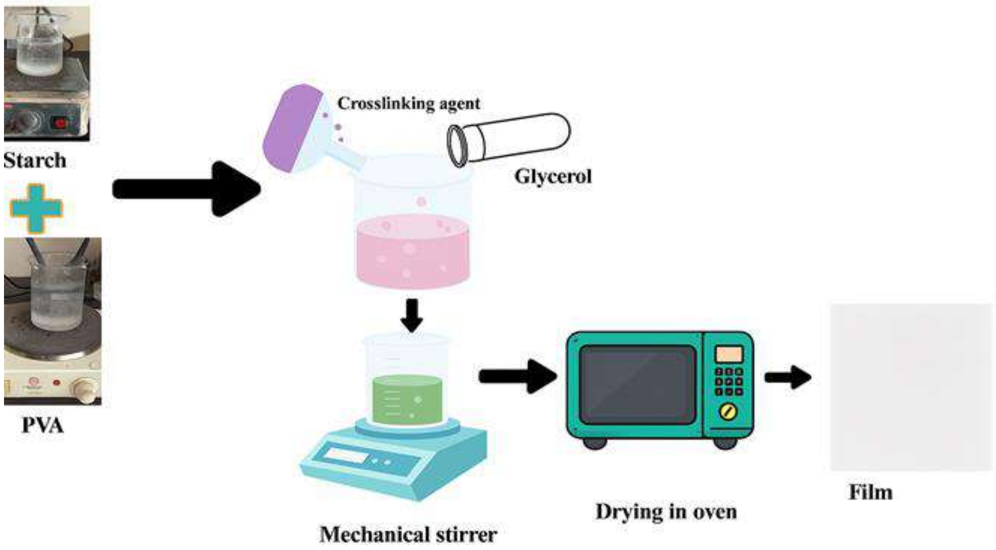


Figure 1: Preparation of film

III. CHARACTERIZATION AND TESTING

A. Mechanical Properties

The Tensile properties of the film were used to measure with UTM Machine in accordance with ASTM D882.

B. FTIR Analysis

The chemical structure and functional group of prepared film were recorded at room temperature on a diamond disc in the 4000-400cm-1 range using a FTIR-Spectrometer Model No: LI-5500.

C. XRD Analysis

The crystallinity of film was analyzed using an The Rigaku Mini XRD with Cu kα radiation (60KV, 50mA) at a wavelength of 1.5406A°.

D. Morphological Analysis

The surface structure of film was analyzed using Sem (Model: EVOS M5000 LV, Make: Italy), with a 12 KV high tension voltage. Prior to imaging, the samples were coated with gold and conditioned for one hour.

E. TGA Analysis

The thermal degradation behaviour of film in the temperature range 50-600°C was examined using Hitachi thermal analyzer in a nitrogen atmosphere at a heating rate of 10°C/min.

F. Biodegradable test

Soil burial method is used to study the natural state. To investigate the biodegradability of film, cut 5 x 5 cm2 of every sample and put it in a compost-prepared pot. The pot spends 14 days in the environment. The sample should be cleaned and dried in an oven after the predetermined amount of time. The provided formula was used to determine the specimen's weight loss percentage:

Weight loss = $[(S_0 - S_1)/S_0] \times 100\%$
where,
S_0 = Initial weight of film before
S_1 = Final weight of film

IV. RESULTS OF STARCH

A. Yield

Starch is extracted with ≅11.6% yield [40].

B. FTIR Analysis

The potato starch (PS) was characterized by FTIR which is shown in figure 2. The study was performed between 4000 to 500 cm-1. The C-O stretching vibrations are responsible for the strong broad band that can be seen in this graph with three peaks at 985, 1089, and 1164 cm-1 (the finger print region). C-O stretching vibration are the most common characteristics for polysaccharides [8]. The peak seen at 3401cm-1, is second characteristic band, called stretching vibration of hydrogen bonded hydroxyl groups. An absorbance peak, found around 715, 767, 858 and 933 cm-1 peak, corresponds to C-O-C ring vibration. C-O and C-H (associated with OH group) stretching are found at peaks 2931 and 1660 cm-1 respectively. An uncommon peak of CO2 was found at peak at 2161 cm-1. At 1465 and 1376 cm-1 are called CH2 symmetric scissoring and C-H symmetric bending respectively [9].

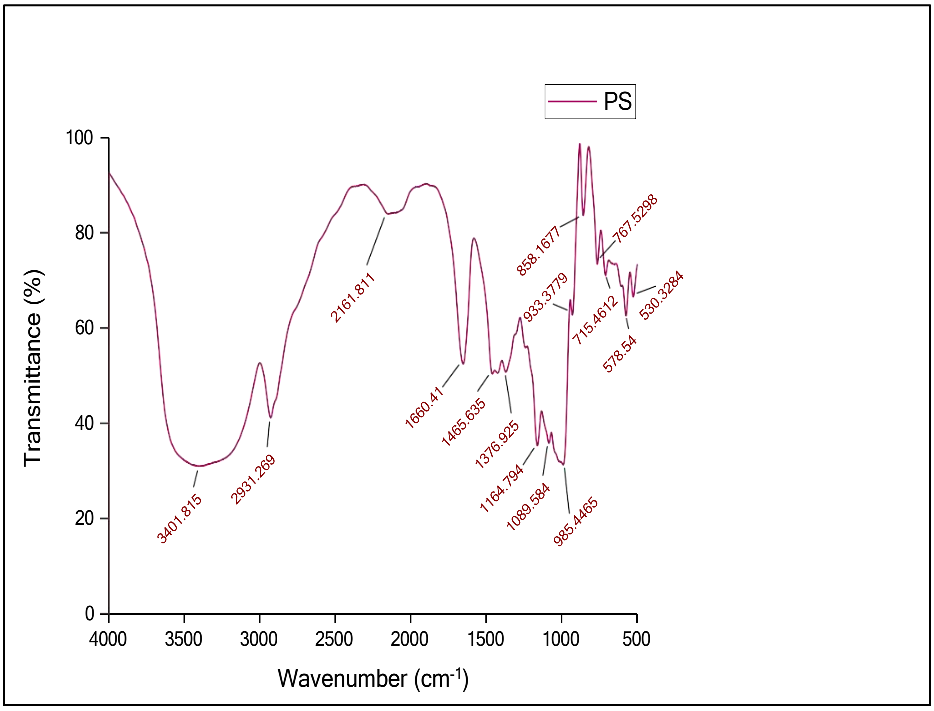


Figure 2: FTIR spectra of potato starch

C. Thermogravimetric Analysis (TGA)

The thermal behavior of the film was analyzed using thermogravimetric analysis (TGA) in the temperature range of 50–600 °C under a nitrogen atmosphere (Figure 3). The first weight loss of 15.05% was observed between 50–200°C, attributed to the evaporation of residual moisture. A major weight loss of 78.9% occurred between 299–598 °C, corresponding to the thermal degradation of potato starch [10].

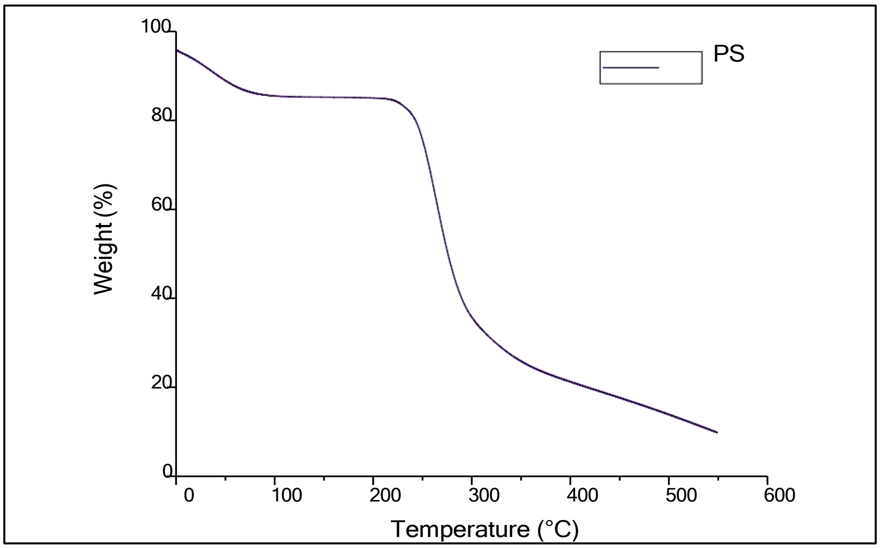


Figure 3: TGA graph of Potato starch

V. RESULTS OF SUCROSE AND OXIDIZED SUCROSE

FTIR Analysis- The sucrose (S) and oxidized sucrose (OS) were characterized by FTIR which is shown in figure 5. Between 4000 and 400 cm-1, the fingerprint region distinct patterns for both sucrose and oxidized sucrose. The hydroxyl groups are indicated by a band in both figures that is located approximately 3400 cm-1. In sucrose, there is a C-H stretching band that measures between 2700 and 2900 cm-1. Around 1621 cm-1 in the graph of oxidized sucrose is indicating stretching vibration of aldehyde groups (C=O) [11].

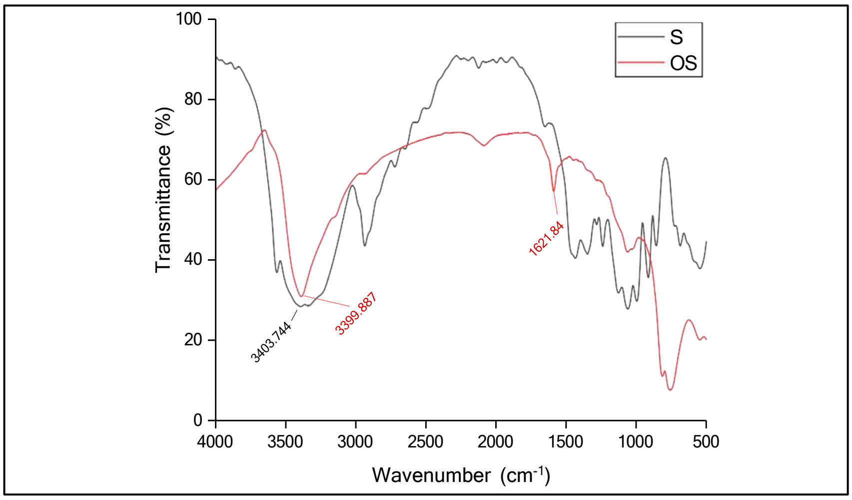


Figure 4: FTIR analysis of Sucrose & Oxidized Sucrose

VI. RESULTS OF STARCH / PVA FILMS

A. FTIR analysis of uncross linked and crosslinked films

Uncross linked film (S5) and crosslinked film (S6) were characterized by FTIR which is shown in fig.5 The area between 1200 and 800 cm-1 designated to the primary, secondary, and tertiary alcohols' C-O stretching vibrations. These all three peaks are being found between 1200 to 1050 cm-1. It can be seen only in crosslinked film's FTIR spectra. When potato starch reacts with oxidised sucrose, the result is the production of ether/acetal linkages, which causes the peaks between 1200 and 1050 cm-1, i.e., C-O stretching vibrations, to get larger. At 1659 cm-1 in crosslinked film belongs to C=C bond. The presence of a hydroxyl group is indicated by peaks between 3500 and3000 cm-1. C-H stretching vibration has been found at peaks 2931 and 2936 cm-1 in S5 and S6 spectra respectively [12][13].

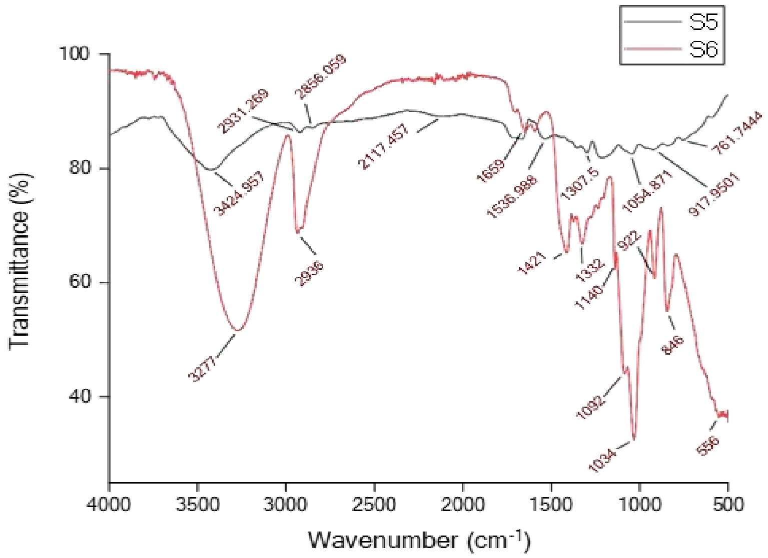


Figure 5: FTIR Spectra of film

B. *XRD Analysis:*

The patterns obtained for S5 and S6 film samples from XRD analysis are shown in the fig.6. In S5 XRD pattern having crystallinity 42.3%, diffraction peaks were observed at 6.7º, 13.8º, 18.4º, 20.1º, 30.8º and 40.8º (2θ). The orderly arrangement of PVA molecules in the films may be responsible for the peak at 20.1º. The diffraction peak around 18.4º shows the presence of starch content [14]. In XRD pattern of S6 film having crystallinity 42.4%, diffraction peaks were found at 6.3º, 16.4º, 20.4º, 30.6º and 41.6º (2θ). In this pattern, peak at 20.4º shows regular arrangement of PVA molecules. The starch molecules might have lost their crystallinity on crosslinking. The peak at 16.46º shows the starch presence in the film [15].

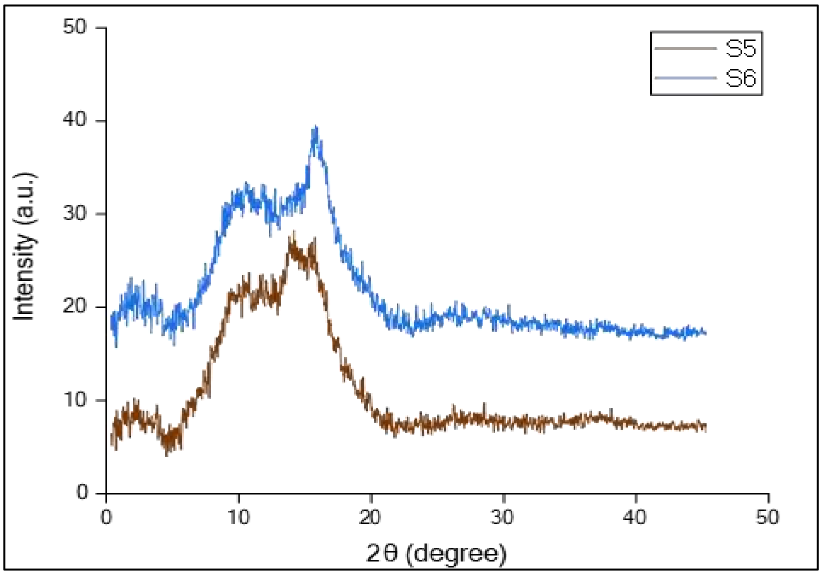


Figure 6: XRD Spectra of film

C. *Mechanical Properties*

As shown in Figure 7, uncrosslinked films (S1, S3, S5) exhibited increasing tensile strength and elongation at break with higher PVA content, with S5 showing the highest values (10.04 MPa and 267%). Similarly, crosslinked films (S2, S4, S6) demonstrated improved mechanical performance due to crosslinking, with S6 recording the maximum tensile strength (13.32 MPa) and elongation at break (357.59%). These results indicate that both increasing PVA content and incorporation of crosslinker significantly enhance film strength and flexibility, with S5 (uncrosslinked) and S6 (crosslinked) exhibiting the best overall properties [16][17][18].

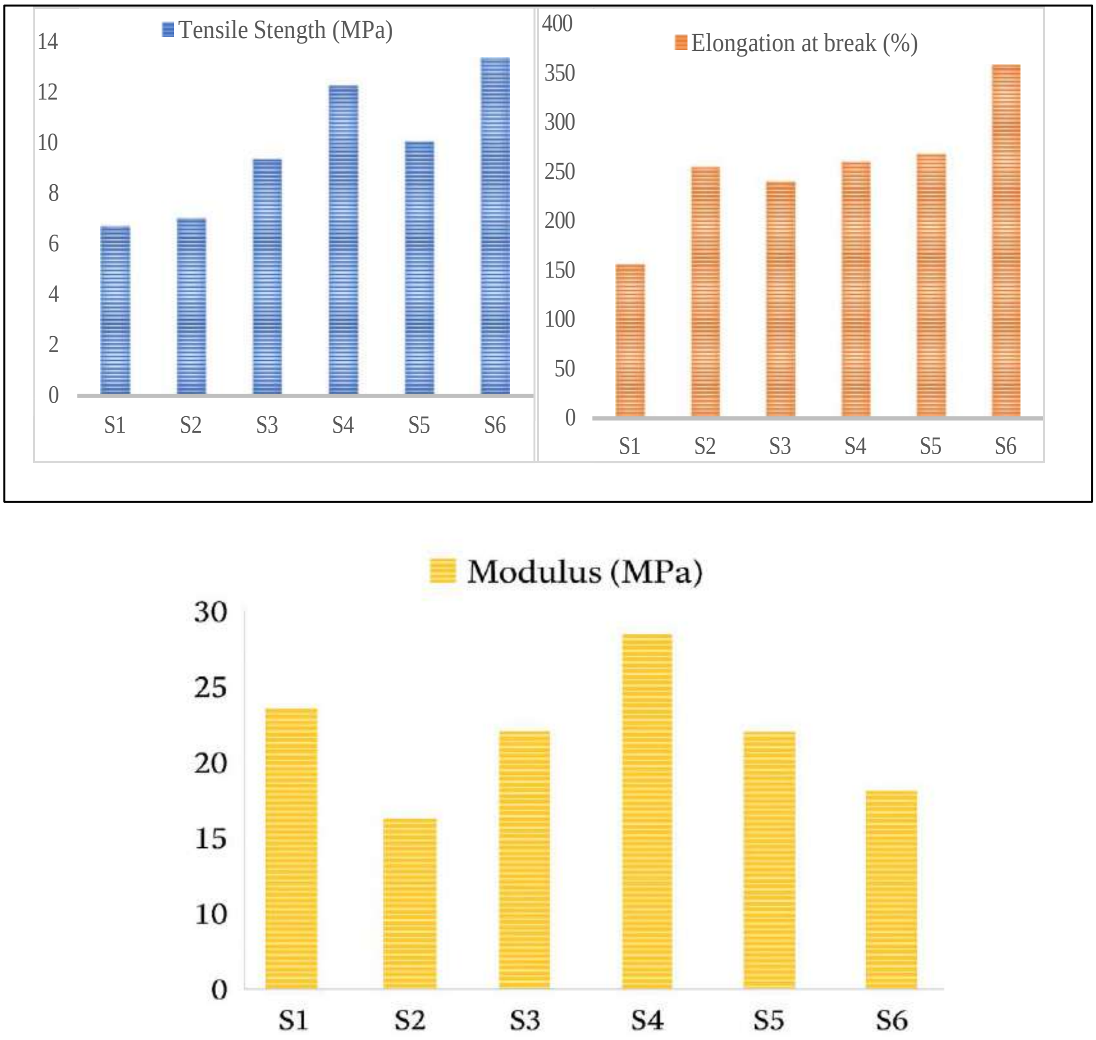


Figure 7: Tensile Properties of film

D. Thermogravimetric Analysis

Figure 7 illustrates the thermal degradation of uncrosslinked (S1, S3, S5) and crosslinked (S2, S4, S6) films, showing four-step degradation. The initial 7–10% weight loss (49–145°C) corresponded to moisture evaporation. Subsequent losses (22–33% and 29–53%) occurred between 120–277 °C and 240–417 °C, attributed to PVA side-chain and starch main-chain degradation, with PVA decomposing faster than starch. Crosslinked films showed higher weight loss in the second stage, indicating the influence of crosslinking bonds. The final 14–20% loss (400–597 °C) was due to carbonization [19][20].

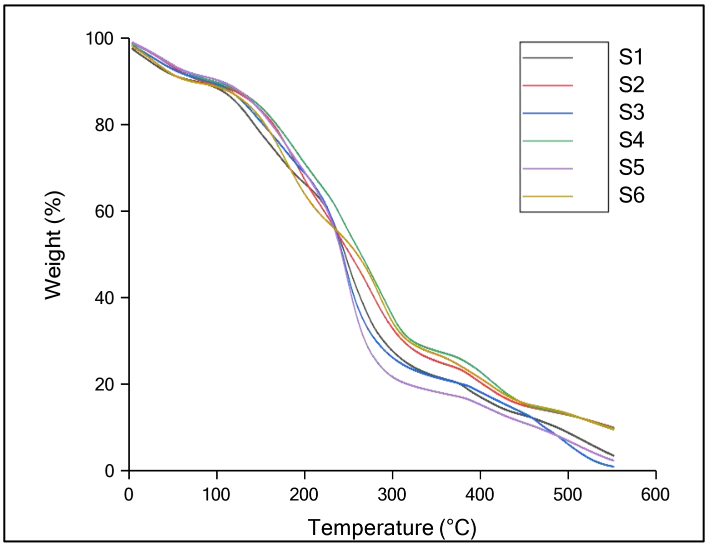
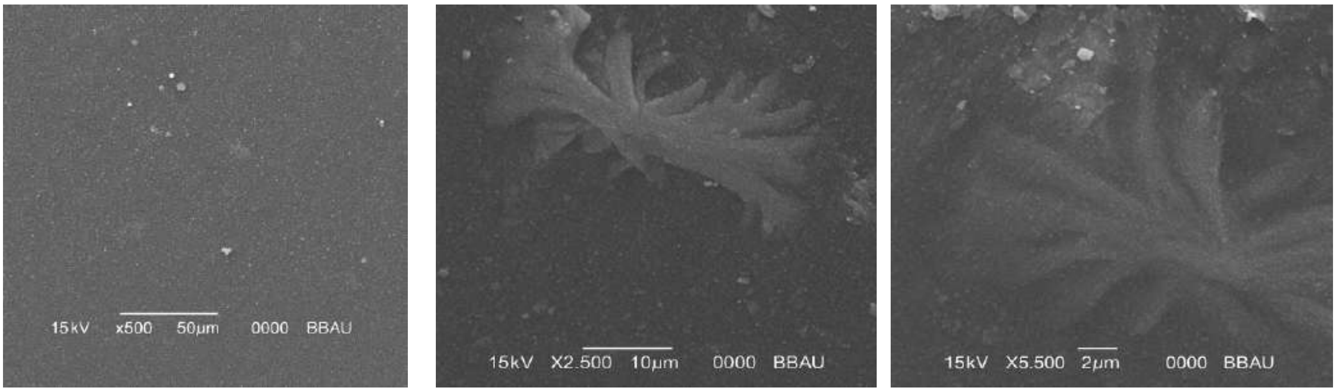


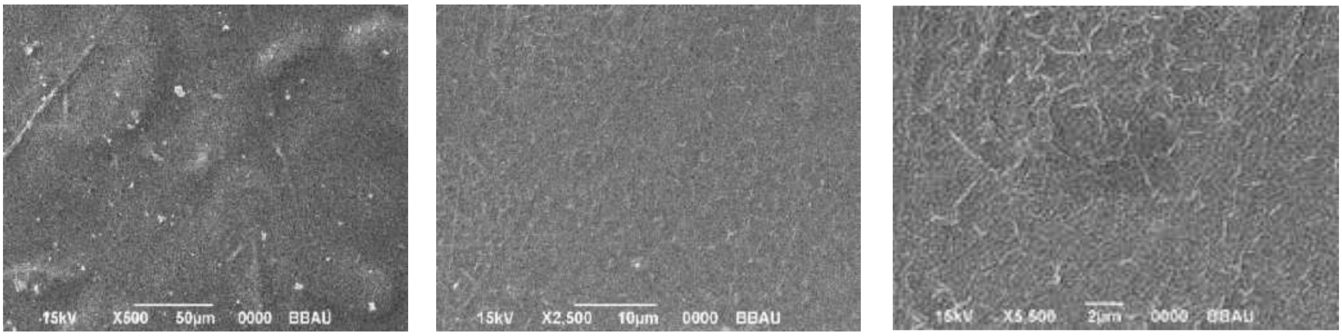
Figure 8: TGA analysis of film

E. SEM Analysis:

The optimized samples of uncrosslinked and crosslinked films were used for SEM analysis to study the morphological properties. The study was done at 500X, 1500X and 2000X as shown in figure 9 There are some agglomerates and granules observed in uncrosslinked film, indicating low compatibility between PVA and potato starch. In contrast to crosslinked films, the crosslinked film has a smooth surface look without any voids and exhibits better adhesion since the crosslinker reduced enhanced the polymer chains' flexibility and the intermolecular forces [21]. The white spots on the crosslinked film morphology indicate the presence of untreated crosslinker.



(a) Uncrosslinked film



(b) Crosslinked film

Figure 9: SEM analysis of film

F. Biodegradability Test

Biodegradability test has been done by soil burial method. In this method degradation occurred by the action of microbes & enzymes. Evaluation of % weight loss of the films was made on 0 & 14 days. As per given data in table.1 % weight loss in the uncrosslinked films (S1, S3, S5) is more as compared to crosslinked films (S2, S4, S6). This is because the crosslinked films have less hydroxyl group and more intramolecular force as compare to uncrosslinked films illustrated in figure 10 [21].

Table 1: (%Weight loss in biodegradability test)

Sample	**% Weight loss**
S1	**44.10**
S2	**40.18**
S3	**37.66**
S4	**30.35**
S5	**44.20**
S6	**50.36**

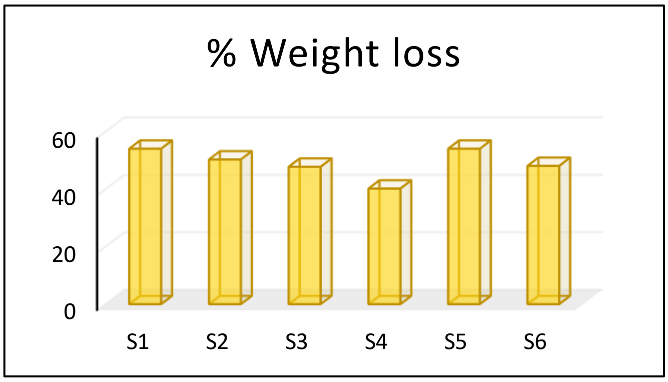


Figure 10: Biodegradability analysis of film

VII. CONCLUSION

Starch was successfully extracted from potato and characterized using FTIR and TGA to verify its purity and structural integrity. Sucrose was effectively oxidized via periodate cleavage to serve as a crosslinker, with FTIR confirming the modification. Films were prepared by solution casting using varying ratios of starch (1–2 g) and

PVA (5–6 g), crosslinked with oxidized sucrose at concentrations of 0.5–1.5%. SEM analysis revealed that crosslinked films exhibited smoother surfaces and improved flexibility compared to uncrosslinked films. Mechanical strength and thermal stability of the films increased with higher crosslinker content, as confirmed by tensile testing and TGA. This study demonstrates a simple, cost-effective, and non-toxic approach to enhance the properties of starch-based PVA/starch films for potential packaging applications.

CONFLICTS OF INTEREST

The authors declare that they have no conflicts of interest.

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