

**A development of biodegradable eco-films using CMC-PVA and Triacetin as
bioplasticizer**

Pranit Patil^a, Ananda J. Jadhav^{*,b}, Dipak V. Pinjari^{*,c}

^a Chemical Engineering Department, Institute of Chemical Technology, Mumbai (India).

^b Biological Sciences and Biotechnology Department, Institute of Chemical Technology,
Mumbai (India).

^c Polymer & Surface Engineering Department, Institute of Chemical Technology, Mumbai
400 019, India

*Author to whom correspondence should be addressed

1. Dr. Dipak V. Pinjari, email: dv.pinjari@ictmumbai.edu.in; dpinjari@gmail.com ; cell phone:
+91-9967719496.
2. Dr. Ananda J. Jadhav, email: aj.jadhav@ictmumbai.edu.in; anandjjadhav@gmail.com ; cell
phone: +91-8928530865.

Abstract

The growing concern over environmental pollution caused by conventional plastic materials has led to an increased interest in the development of biodegradable and non-retrogradable eco-films as potential alternatives. This research focuses on the fabrication and characterization of eco-films based on Carboxymethyl cellulose (CMC) and Polyvinyl Alcohol (PVA) with Triacetin as a bioplasticizer. The objective is to investigate the mechanical, thermal, and biodegradable properties of the resulting films and assess their potential for various applications. The blended PVA/CMC films were formulated using different Triacetin composition (5, 10, 15, 20, 25 and 30 wt. %). The formulated film was characterized using Fourier transform infrared spectroscopic analysis and mechanical properties such as tensile strength, elongation, thickness have been measured. The biodegradability rate of the film was also investigated to confirm its biocompatibility. The Triacetin impart good plasticity, which effects on structure and properties of CMC-PVA film. The elongation and tensile strength of the crosslinked films were much higher than the non-cross linked films, having the same composition. The crosslinked film possesses higher moisture content than non-crosslinked film at a same concentration of Triacetin.

Keywords: Triacetin, Biodegradable Film, Polyvinyl alcohol, Carboxymethyl cellulose.

1. Introduction

In response to the escalating levels of water, land, and air pollution, there has been a growing demand for eco-friendly products labeled as biodegradable, environment-friendly, and non-toxic. This need has become crucial as "White Pollution" continues to surge due to rapid development and industrialization. The extensive use of petroleum-based materials has played a significant role in global environmental degradation, and the depletion of non-renewable petroleum sources has become a pressing concern. To address these issues, there is a compelling need to explore renewable sources and materials that can easily degrade without harming the environment [1]

The recurring problem of plastic disposal arises from its slow degradation process. The excessive accumulation of plastic waste has caused severe environmental issues, and it contributes to landfill depletion because of its high volume-to-weight ratio and resistance to breaking down over time. Non-biodegradable plastics, which are derived from petroleum-based products, possess high resistance to microbial degradation. As a result, these plastics persist in the environment and their accumulation continues to grow. To address this issue, researchers have developed biodegradable plasticizers that do not harm our ecosystem, offering a potential solution to the problem [2][3]

Biodegradable polymers belong to a loosely defined group of polymers that can be broken down by living organisms. They present a potential alternative to conventional non-biodegradable polymers [4] [5]. In recent times, substantial advancements have been made in creating biodegradable products using agricultural raw materials. These biodegradable polymers can be categorized into synthetic and natural types, with the natural ones further divided into plant and microbial origins [6]. The degradation of such polymers involves breaking them down into their constituent monomers. This requires the presence of unstable and hydrolyzable linkages that facilitate chemical, biological, or photochemical reactions [7]

Among the well-known and essential examples are polyhydroxy fatty acids and polyhydroxyalkanoates (PHA), which are polyesters produced and accumulated by bacteria as energy storage or reserves.

Among the various polymers utilized in the development of biodegradable films, starch has garnered significant attention, as reported.[8] Starch is a natural polymer derived from renewable resources, widely available, and cost-effective. It possesses the unique ability to create a continuous thermoplastic material [9][10][11] Carboxymethyl cellulose (CMC) also growing importance due to its remarkable property, such as high viscosity, is non-toxic, and is non-allergic. CMC is a cellulose ether obtained by carefully reacting alkali cellulose with sodium mono-chloroacetate under precisely controlled conditions. It possesses many desirable qualities, such as thermal gelation, filming, emulsification, binding and inspissation [12]. The abundance of hydroxyl and carboxylic groups in CMC facilitates its water-binding and moisture sorption capabilities. Additionally, CMC exhibits excellent biodegradability and low cost, making it highly suitable for various applications [13]. Due to its polymeric structure and high molecular weight, CMC has been utilized as a filler in biocomposite films. Biofilms can indeed be prepared using CMC, although the strength of the resulting product may pose a challenge. However, a solution to this issue lies in blending CMC with polyvinyl alcohol (PVA) since CMC and PVA are compatible. By incorporating PVA into the blend, the film's strength can be improved, leading to a more favorable end product. PVA is a water-soluble, non-toxic synthetic polymer commonly used as an engineering material. Incorporating PVA improves the material's thermal and mechanical properties by altering the polymer structure at both the molecular and morphological levels. This modification leads to enhanced performance characteristics in the resulting material.

Bioplasticizers used in biodegradable films are typically derived from renewable resources, making them more environmentally friendly compared to traditional plasticizers derived from

petrochemicals. Triacetin is one of the bio-additives which is synthesized by acetylation as well as esterification reaction [14][15][16] Triacetin serves as a plasticizer for cellulosic resins and is fully compatible with cellulose acetate, nitrocellulose, and ethylcellulose. It enhances plasticity and flow in laminating resins, particularly at low temperatures, and also functions as a plasticizer for vinylidene polymers and copolymers. Bioplastics utilized in food packaging often incorporate glycerol [17] and sorbitol [18] as plasticizers. In order to safeguard perishable foods, antimicrobial agents have been employed [19]. Triacetin possesses the dual benefits of functioning as a plasticizer and offering antimicrobial and antifungal properties, making it a potential substitute for conventional plasticizers and antimicrobial agents in food packaging [20][21]

The aim of the paper is to create and analyze environmentally friendly films by combining CMC and PVA, using Triacetin as the bioplasticizer. To achieve this, the films underwent crosslinking with glutaraldehyde. The study was primarily focusing on evaluating crucial parameters, such as moisture content, tensile strength, elongation at break, and biodegradability of the films that were produced as a result of this process.

2. Experimental

2.1 Materials

Sodium Carboxy Methyl Cellulose (CMC) and partially hydrolyzed (Cold) Poly Vinyl Alcohol (PVA) were obtained from SD Fine Chemicals Ltd, Mumbai, and served as the fundamental polymers for film preparation. Glutaraldehyde, which was also acquired from SD Fine Chemicals Ltd, Mumbai, was utilized for crosslinking the films. Triacetin of analytical reagent (AR) grade was procured from Thomas Baker (Chemicals) Pvt. Ltd, Mumbai, and served as the bioplasticizer in this study.

2.2 Preparation of paste for casting using CMC-PVA blend

In the manufacturing process of these films, a 150g batch consisting of a 5% paste acted as the primary matrix, augmented with diverse amounts of a plasticizer (5, 10, 15, 20, 25, and 30 wt% of the film) according to specifications detailed in Table 1. It is noteworthy that the employment of a 10% CMC/PVA solution posed difficulties in attaining consistent film formation owing to its heightened viscosity. Subsequent modifications were introduced in the paste formulations to enhance the efficiency of the film manufacturing procedure.

Carboxymethyl cellulose was accurately measured and dissolved in 50 grams of water. Likewise, appropriate quantities of PVA and the specified plasticizer were weighed and dissolved in 50 grams of water. Subsequently, the CMC solution was combined with the latter solution. This amalgamated solution underwent heating at 90°C for 45 minutes, with continuous stirring facilitated by an overhead stirrer. Following this heating phase, a 15-minute interval was allocated for the solution to settle, minimizing the formation of foam resulting from the vigorous stirring process. To maintain a consistent concentration of the final paste, the solution's volume was adjusted to 150 milliliters by adding water. This solution showcased favorable flow consistency and viscosity, which are pivotal for the successful production of the films. Refer to Table 2, for a comprehensive breakdown of the formulations employed in the creation of various films.

2.3 Preparation of paste for hydrophobic films

To formulate the hydrophobic film, the procedure entailed adding glutaraldehyde at a concentration of 10% by weight relative to the total paste. This crucial step facilitated crosslinking reactions between CMC and PVA polymers. The paste underwent stirring for a duration of 3 hours. Subsequently, the films underwent conditioning at room temperature for 12 and 24 hours before commencing the testing phase. This conditioning period plays a

pivotal role in ensuring the attainment of precise and dependable results. A detailed breakdown of the ingredients utilized for each variation is available in Table 3.

2.4 Biodegradable film characterization

2.4.1 Tensile strength and elongation at break

The assessment of film strength followed the guidelines outlined in ASTM D 5035(1995), the standard test method for evaluating breaking strength and elongation of films. Testing was conducted using a Tinius Olsen H5KS universal testing machine (UTM) designed specifically for tensile strength analysis. Primarily, this method encompasses the cut strip and ravel-strip techniques, with the present study employing the cut-strip procedure to determine film strength.

The specimen used for testing had dimensions of 25 mm in width and 150 mm in length. During testing, specific parameters were employed: a gauge length of 50 mm was utilized, and the actual testing width corresponded to 25 mm. The loading rate applied during the test was set at 100 mm/min. The instrument used enabled the determination of both breaking force and elongation at the point of breakage for this specimen.

2.4.2 Film thickness

The thickness was calculated as the average of 10 measurements taken with a hand-held Digital Vernier Caliper (Mitutoyo CD 6" CS model) with a minimum count of 0.01 mm for each sample.

2.4.3 Moisture content (MC)

The moisture content (MC) was computed following the procedure outlined by [22]. Film samples were initially weighed (W_0) into glass dishes, then subjected to drying for four hours in an air-circulating oven at 105°C. Subsequently, the dried samples were reweighed (W_i). The moisture content of each film was calculated using the equation, and the process was

conducted in quadruplicate to ensure accuracy in the determination of moisture content for all film samples.

$$MC = \frac{(W_0 - W_i)}{W_0} \times 100 \quad (1)$$

2.4.4 FTIR analysis

An FTIR-8400S IRAffinity-1 Fourier Transform Infrared Spectrophotometer manufactured by Shimadzu, Japan, was employed for the analysis. Each test utilized 0.1 grams of the sample. Before the IR tests, every sample was vacuum-sealed in a water-resistant polyethylene pack and securely stored within a silica gel desiccant container to maintain its integrity and shield it from moisture exposure.

2.4.5 Biodegradation test

The specimens were cut into 2 cm × 2 cm pieces. Vegetable compost, employed as soil, underwent sieving to eliminate sizable clumps and was subsequently poured into a plastic tray, reaching a depth of approximately 6 cm. The samples were buried under 4 cm of soil within these conditions: ambient temperature (~25°C) and humidity levels ranging between 70–80%. To maintain adequate moisture, the compost received regular misting twice daily. At various intervals, the films were extracted from the soil and subjected to drying in a vacuum oven at 50°C for 24 hours, following the method described by [23].

3 Results and discussions

The resulting films exhibited a transparent, glossy appearance and were notably easy to handle, devoid of any visible bubbles. Notably, films lacking plasticizers displayed a brittle nature, whereas those incorporating plasticizers showcased enhanced flexibility and malleability. Specifically, the introduction of Triacetin not only augmented visual thickness but also preserved the transparency of the bioplastics. Remarkably, the preparation of CMC-

PVA films proved to be straightforward, necessitating no gelatinization process and enabling drying at lower temperatures, facilitating ease of production.

3.1 Tensile strength, elongation at break and thickness of CMC-PVA films

PVA, carboxy methyl cellulose, and starch are categorized as polar polymers, suggesting that their composite could yield a material boasting exceptional mechanical properties (Taghizadeh, Sabouri, and Ghanbarzadeh, 2013). The impact of a plasticizer on tensile strength and elongation at break is contingent upon various factors, including the inherent nature of the polymer and plasticizer, the quantity of plasticizer employed, and the conditions under which it integrates into the system.

As depicted in Table 4, the mechanical characteristics of CMC-PVA films portray an intriguing pattern. Tensile strength exhibited an upward trajectory in tandem with increased plasticizer concentration [24], resulting in higher tensile strength values. Correspondingly, elongation at break also demonstrated an increase correlated with higher plasticizer concentrations (Lin and Ku, 2008). Notably, the film incorporating 30% triacetin with a CMC-PVA ratio of 2.5:2.5 showcased the highest values for both tensile strength and elongation at break compared to other films. Optimal mechanical properties were realized at a CMC-PVA ratio of 2.5:2.5 with 30% Triacetin.

Table 5, illustrating the tensile strength and elongation at break for crosslinked films, a similar trend emerges. Elevated plasticizer concentration corresponded to increased tensile strength, particularly noticeable at higher concentrations. Similarly, elongation at break followed a comparable pattern, exhibiting higher values with increased plasticizer concentrations within non-crosslinked films. Notably, crosslinked films displayed superior tensile strength and elongation at break in comparison to non-crosslinked films. The pinnacle of both tensile strength and elongation at break was observed with a concentration of 30%.

These findings underscore the correlation between plasticizer concentration, film composition, and resultant mechanical properties. Notably, a significant enhancement in both tensile strength and elongation at break was evident in films featuring higher plasticizer concentrations, particularly at 30%, suggesting the potential for optimizing mechanical properties through precise adjustments in plasticizer content and film composition."

3.2 Moisture content (MC)

The moisture content analysis of CMC-PVA films, detailed in Table 6, revealed levels ranging between 6-14%. A discernible correlation emerged, demonstrating that an escalation in plasticizer concentration corresponded to an increase in moisture content across all films. Moreover, the specific type of plasticizer employed exerted a noticeable influence on moisture levels. Notably, it has been documented that CMC exhibits cold water solubility, and the moisture absorption capacity of CMC powder stands at approximately 13%, surpassing that of PVA. Consequently, the elevation in CMC concentration within the blend contributed to an escalated moisture content in the resultant films.

Further elucidation can be found in Table 7, depicting moisture content for hydrophobic films, where a consistent pattern akin to that observed in non-crosslinked films was identified. However, it is noteworthy that the moisture content of hydrophobic films surpassed that of their non-crosslinked counterparts.

This data emphasizes the direct influence of plasticizer concentration, the type of plasticizer, and the composition of the film on moisture content. Specifically, the inclusion of CMC, known for its heightened moisture absorption capacity, played a pivotal role in augmenting moisture content in the films, thereby contributing to an understanding of the intricacies governing moisture uptake in CMC-PVA films.

3.3 FTIR Analysis

The FTIR spectra depicted in Figure 2a and Figure 2b illustrate the differences observed in CMC–PVA blend films with and without the presence of a plasticizer. An analysis of the impact of the plasticizer was conducted by comparing the spectra of films containing plasticizer to those lacking it. Notable characteristic bonds were identified in these spectra, including a peak at 3358 cm⁻¹ representing the stretching of the OH group associated with CMC, triacetin, and water; another range at 2880–2900 cm⁻¹ attributable to CH stretching; and a peak at 1645 cm⁻¹ assigned to the water adsorbed by CMC molecules.

The comparison of available OH groups in different systems involved assessing the ratio between the intensity of the peak at 3358 cm⁻¹ and that at 1095 cm⁻¹, related to the stretching vibration of (C-O) in the (C-O-H) group. Interestingly, the intensity of the 1095 cm⁻¹ and 3358 cm⁻¹ peaks in CMC-PVA films with plasticizer exceeded that of films without the plasticizer, suggesting a reduced number of available OH groups. The shift in spectra to higher values following the addition of plasticizers indicated a promotion of hydrogen bonding interactions between CMC and plasticizers. This alteration was further substantiated by the increased intensity and sharpness of the peak, providing insight into the augmented strength of the hydrogen bonding.

Overall, these findings point towards the significant impact of plasticizers on the molecular interactions within CMC-PVA films, leading to the enhancement of hydrogen bonding, as indicated by alterations in peak intensity and position in the FTIR spectra.

3.4 Biodegradability test

The pivotal properties defining biodegradable materials encompass water absorption capacity and degradability. In the case of the PVA:CMC blend film, the water absorption capacities exhibited significant variability, aligning consistently with findings previously reported (Taghizadeh, Abbasi, and Nasrollahzade, 2012). The soil burial test simulated actual

degradation conditions, showcasing weight loss data outlined in Table 8. The percentage of weight loss directly correlated with the films' degradation. Weight loss measurements were tracked at intervals of 2, 4, 6, 8, and 10 days, revealing degradation rates contingent upon film composition; notably, crosslinked films displayed a slower degradation pace compared to non-crosslinked ones. Impressively, a weight loss of 70-80% was observed within ten days, and beyond this timeframe, weight loss became challenging to measure due to extensive degradation. The swift weight loss observed can be attributed to the films' soluble nature, wherein moisture, a critical factor in microbial growth, played a significant role in facilitating degradation. Extrapolation of the weight loss values and graph analysis suggested complete degradation of the films. Notably, the % weight loss values indicated that these films are readily biodegradable, making them suitable for applications demanding low durability and swift biodegradability. Figures 3a and 3b confirmed these findings, highlighting that the biodegradation rate of non-crosslinked films surpassed that of crosslinked films. This further accentuates the influence of film crosslinking on the degradation process, indicating potential variations in their biodegradation rates.

4. Conclusion

Triacetin, as a plasticizer, wielded a multifaceted influence on the structures and properties of CMC-PVA films. Its integration facilitated enhancement in the C-O linkage of the C-O-C groups within the CMC-PVA film. The increased presence of triacetin molecules expanded the spacing between macromolecular chains and the overall amorphous regions. This enlargement subsequently augmented the free volume and facilitated chain motions within the film matrix.

The enhanced C-O bonding and the constraints imposed by the crystalline structure collectively influenced the mobility of macromolecular chains, thereby enhancing the thermal properties of the matrix. The incorporation of triacetin significantly altered the film matrix

due to dynamic interactions with starch ester molecules. This led to heightened flexibility of the macromolecular chains, exacerbating the reduction in storage tensile modulus upon heating and promoting relaxation across various scales, encompassing side groups, shorter molecular chains, and ultra-nanometer scales.

However, these viscoelastic behaviors were restricted by the crystallite, limiting their extent. The distribution of mobile entities contributing to the glass–rubber transition matrix was influenced by diverse microstructures within the overall structure–property relationship. These intricate changes are anticipated to contribute significantly to further modifications in the structure and potential applications, particularly in varying environmental conditions, such as the processing temperature of hydrophobic biodegradable packaging

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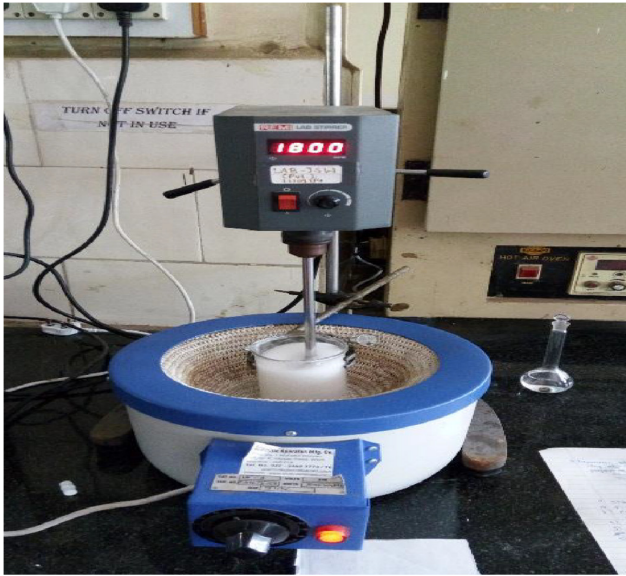
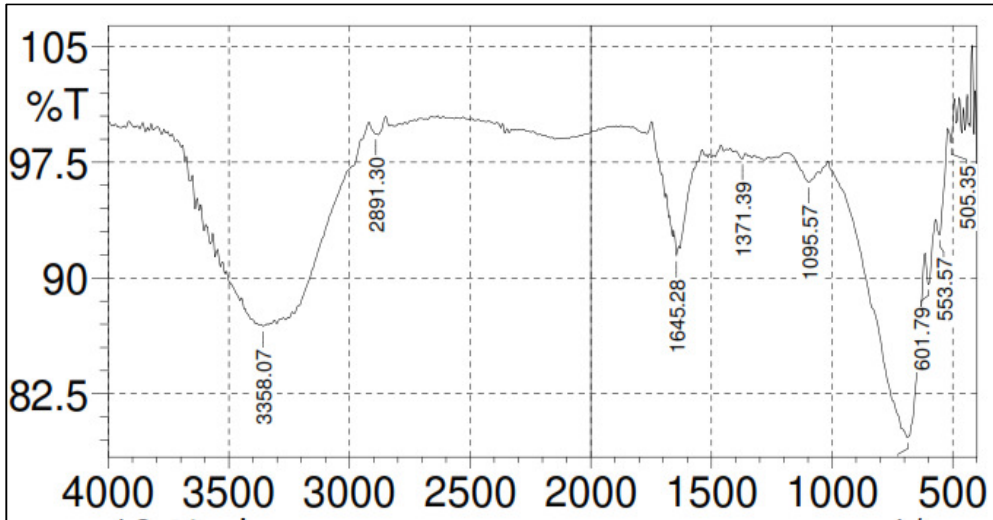
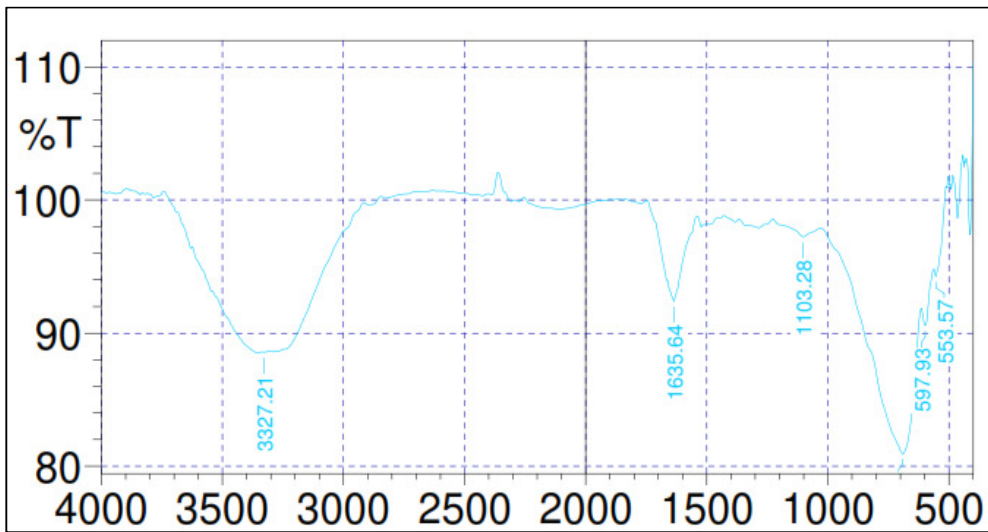


Figure 1: Experimental setup

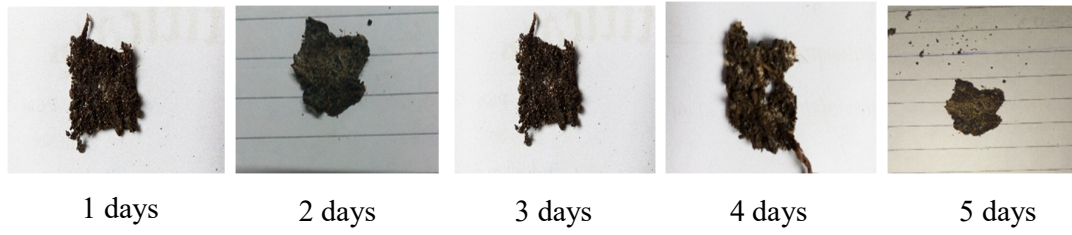


(a)

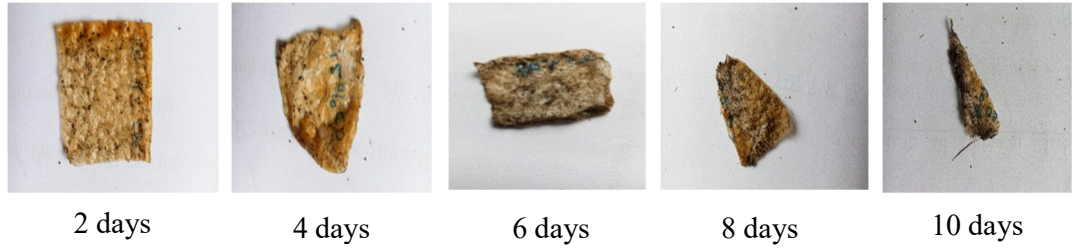


(b)

Figure 2: FTIR Spectra of developed films (a) FTIR Spectra of studied films with plasticizer (b) FTIR spectra of studied films without plasticizer.



(a)



(b)

Figure 3: (a) Biodegradation of non-crosslinked film, (b) Biodegradation of crosslinked film

Table No.1: Sample Composition of different developed systems

ponents	
tin	5,20,25,30 (of total paste)
	ning quantity

Table 2: Sample compositions of the different developed system

CMC: PVA blend	Plasticizer (%)
	0
	5
	10
2.5:2.5	15
	20
	25
	30

Table 3: Sample compositions of different hydrophobic films

CMC: PVA blend	Plasticizer (%)	Glutaraldehyde (%)
	0	
	5	
	10	
2.5:2.5	15	10
	20	
	25	
	30	

Table 4: Tensile strength, elongation at break and thickness of CMC-PVA films plasticized with Triacetin

CMC: PVA blend	Plasticizer (%)	Tensile Strength, (Kgf)	Elongation (%)	Thickness, (mm)
2.5:2.5	0	0.271	0.729	0.02
	5	0.372	0.873	0.02
	10	0.494	1.080	0.02
	15	0.561	1.665	0.02
	20	0.882	2.613	0.02
	25	1.224	3.975	0.02
	30	1.392	4.435	0.02

Table 5: Tensile strength, elongation at break and thickness of crosslinked CMC-PVA films plasticized with Triacetin

CMC: PVA blend	Plasticizer (%)	Glutaraldehyde (%)	Tensile Strength, (Kgf)	Elongation (%)	Thickness, (mm)
	0		0.799	1.502	0.035
	5		1.361	1.578	0.03
	10		1.412	2.679	0.03
2.5:2.5	15	10	1.361	2.920	0.025
	20		1.621	3.712	0.03
	25		1.886	4.420	0.03
	30		2.614	5.022	0.03

Table 6: Moisture content of developed films

CMC: PVA blend	Plasticizer (%)	Moisture content (%)
	0	7.25
	5	8.53
	10	9.1
2.5:2.5	15	10.76
	20	12.10
	25	12.71
	30	13.28

Table 7: Moisture content of developed crosslinked films

CMC: PVA blend	Plasticizer (%)	Glutaraldehyde (%)	Moisture content (%)
	0		8.02
	5		8.89
	10		10.10
2.5:2.5	15	10	11.47
	20		13.25
	25		14.71
	30		15.07

Table 8: Weight loss for non-crosslinked films

Plasticizer (%)	% Weight loss due to degradation after days				
	1	2	3	4	5
0	9	29	48	64	87
5	4	23	48	62	85
10	5	23	45	60	88
15	4	19	46	59	87
20	5	17	40	56	82
25	3	14	35	56	86
30	4	13	32	54	78

Table 9: Weight loss for crosslinked films

Plasticizer (%)	% Weight loss due to degradation after days				
	2	4	6	8	10
0	11	23	42	58	77
5	9	17	44	58	79
10	8	17	38	57	74
15	7	19	36	53	72
20	7	15	36	52	72
25	6	13	32	50	70
30	3	11	29	48	69