

Mechanical, Thermal and Biodegradation Properties of Maleic Anhydride-Coupled Nanocrystalline Cellulose/Zein Bioplastics Derived from Agricultural Biomass

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Nanocrystalline cellulose/zein (NCC/zein) bioplastic films coupled with maleic anhydride (MAH) were prepared from sugar-palm solid waste and corn waste to valorize agricultural biomass into biodegradable film materials. NCC was isolated from sugar-palm solid waste, whereas zein was extracted from corn waste with aqueous ethanol. Films containing 0-5 wt.% NCC were produced by melt intercalation using MAH and characterized by FTIR, ¹H NMR, XRD, tensile testing, TG/DTA, soil burial and SEM. FTIR bands at 3431.51, 2879.85, 1635.71 and 1074.40 cm⁻¹ confirmed hydrogen-bonded OH/NH, C-H, carbonyl and C-O groups in the coupled matrix. After correction of stress units, the highest tensile strength and elongation at break were 3.535 MPa and 10.954% in NCC/zein 0%, while the highest apparent secant modulus at break was 60.02 MPa at 1 wt.% NCC. The maximum biodegradation occurred at 5 wt.% NCC with 2.7287% weight loss day⁻¹. The results support further development of biomass-derived biodegradable films; however, the packaging suitability cannot be concluded without dedicated barrier, water-uptake and surface-wettability measurements.

Keywords: Nanocrystalline cellulose, Zein, Maleic anhydride, Bioplastic, Agricultural biomass, Biodegradation.

INTRODUCTION

Plastics derived from fossil fuels are widely used owing to their low cost, light weight and ease of processing, but their resistance to natural degradation results in persistent solid waste [1,2]. Rising plastic consumption and the finite nature of fossil based resources have stimulated interest in biodegradable and bio-based polymers for packaging applications [3-5]. Biopolymers derived from renewable agricultural feedstocks are attractive alternatives, as they can form functional films while developing value from biomass residues [6-13].

Zein, the major prolamin protein in maize, is a hydrophobic storage protein capable of forming films through intermolecular hydrophobic interactions, hydrogen bonding, and constrained disulfide linkages [14-17]. Previous studies [18-23] have reported zein- and corn-based films prepared by controlling solvent, plasticizer and filler processing conditions. Zein films are generally brittle and require reinforcement or interfacial bonding to improve their mechanical performance and processability.

Nanocrystalline cellulose (NCC) is a promising reinforcing phase for biopolymer matrices owing to its high aspect

ratio, abundant hydroxyl groups, crystallinity and potential compatibility with polysaccharides and proteins [24-33]. NCC can be isolated from lignocellulosic biomass and has been widely investigated for bionanocomposites and food packaging films [6-9,24-36]. Sugar palm residues and corn waste provide locally available sources of cellulose and zein, respectively, particularly in agricultural regions and their valorization offers a route for reducing biomass waste while generating value-added materials.

Maleic anhydride (MAH) is a useful coupling agent that can introduce polar anhydride-derived groups and promote interactions between hydrophilic and hydrophobic phases [37-39]. In NCC/zein systems, MAH coupling may strengthen interactions between hydroxyl-rich NCC and the amino and amide groups of zein, leading to a more integrated bioplastic network. Recent studies have emphasized nanocellulose-based films for barrier applications, active packaging and safety assessment in food-contact materials [6,7,39,40]. Packaging performance, though, requires direct evaluation of properties such as water-vapour permeability, oxygen permeability or transmission, water uptake and surface wettability. These parameters were not examined in the present study; the work

is limited to material structure, mechanical response, thermal stability, biodegradability and morphology and does not claim packaging readiness.

This study describes the preparation of MAH-coupled NCC/zein bioplastic films using sugar palm and corn biomass and evaluates their structure, crystallinity, tensile strength, thermal stability, biodegradability and surface morphology. The study provides a material-chemical basis for the further development of biodegradable films derived from agricultural biomass.

EXPERIMENTAL

Sugar palm solid waste was used as a lignocellulose source for the preparation of nanocrystalline cellulose (NCC), while corn waste from a feed processing stream was used as a source of zein. Maleic anhydride (MAH) was used as a coupling agent. Ethanol, methanol, toluene, hydrochloric acid, sulphuric acid, formic acid, acetic acid, hydrogen peroxide, calcium hydroxide, sodium borohydride, aluminum chloride and dimethylacetamide were used unchanged for extraction, hydrolysis, washing and film preparation. α -Amylase was used during the pretreatment of corn waste. Effective microorganisms (EM4) were used in the soil burial test for biodegradation.

Characterization: The principal instruments were an FTIR spectrometer (Shimadzu 8400), ^1H NMR/ ^{13}C NMR spectrometer (Delta 2 ECA, 500 MHz), X-ray diffractometer, TG/DTA device (Seiko), scanning electron microscope (JEOL JSM-6360), heat press, laboplastomill and universal tensile strength tester. Tensile tests were performed according to the general procedure of ASTM D638 for plastic tensile strength properties.

General procedure: Nanocrystalline cellulose (NCC) was prepared from sugar palm solid waste by drying, hydrolysis, decanting, centrifugation, neutralization and a final drying step. Zein was extracted from corn waste after the material was cleaned, ground and steamed with α -amylase at 100–150 °C for 2 h. The treated material was dried, ground to a particle size of 100 mesh, and extracted with 95–100% ethanol at 65–70 °C for 3 h. The solvent was removed by decanting and evaporation to obtain solid zein. The extraction procedure yielded a zein yield of approximately 14% (w/w).

MAH-coupled NCC/zein bioplastics were produced by melt intercalation. NCC, zein and MAH were weighed out according to the designed composition. The processing chamber was preheated to 180 °C for 30 min to stabilize the temperature. The polymer phase was melted at 180 °C, after which zein and NCC were gradually added under continuous stirring. Subsequently, MAH was added and the mixture was mixed for another 10 min. The hot mixture was removed from the chamber, placed on aluminum foil and cooled to room temperature. The film series was coded as NCC/zein 0, 1, 2, 3, 4, and 5%, where the number indicates the NCC content in the NCC/zein mixture.

Detection method: Chemical interactions were examined using FTIR and ^1H NMR spectroscopy. Crystalline structure was analyzed by XRD over a 2θ range of approximately 10–50°. Tensile strength and elongation at break were measured

using dumbbell-shaped specimens. Tensile stress was normalized to the initial cross-sectional area and instrument readings were converted from kPa to MPa. Since the available mechanical dataset comprised ultimate tensile stress and elongation at break rather than the initial linear stress–strain slope for each specimen, the parameter previously reported as Young's modulus was recalculated as the observed intersecting modulus at break:

$$E_{\text{sec}} = \frac{\sigma_b}{\epsilon_b} \times 100$$

This value was used as a comparative stiffness index and does not represent the initial-slope Young's modulus. Thermal stability was assessed by TG/DTA using the initial, maximum and final temperatures of the first and second decomposition stages, along with the residual carbon at 490 °C.

Biodegradation studies: Biodegradation was assessed by burying the samples in garden soil enriched with EM4 for 60 days under indoor conditions away from direct sunlight. Five specimens were used for each formulation. The samples were weighed at 7-day intervals and weight loss was expressed as percentage loss per day. Selected specimens before and after degradation were examined by SEM to assess changes and damage to the surface morphology.

RESULTS AND DISCUSSION

Film formation and chemical structure: Nanocrystalline cellulose (NCC) was obtained as a fine brown powder since the biomass was processed without bleaching. Zein extracted from corn residues appeared as a yellowish-brown organic solid. The NCC/zein films were continuous and could be handled as thin sheets. Increasing NCC content resulted in a rougher surface and darker brown colour, consistent with the higher contribution of unbleached lignocellulosic components to the film appearance.

The FTIR spectra of NCC, zein and MAH-coupled NCC/zein bioplastic are shown in Fig. 1, with the band assignments listed in Table-1. Broad bands at 3441.16–3416.08 cm^{-1} for NCC and zein and at 3431.51 cm^{-1} for the bioplastic were assigned to O–H/N–H stretching vibrations associated with hydrogen bonding [16,17,29–33]. The C–H stretching bands occurred at 2902.99, 2877.92 and 2879.85 cm^{-1} for NCC, zein and the bioplastic, respectively. The carbonyl band shifted from 1645.00 cm^{-1} in NCC and 1652.10 cm^{-1} in zein to 1635.71 cm^{-1} in the bioplastic. Changes in the carbonyl and C–O-related bands are consistent with interactions between NCC and zein within the MAH-coupled matrix [24–27,37–39]. The ^1H NMR spectrum contained signals attributable to R–H, R–OH and R–NH₂ environments, supporting the presence of cellulose- and protein-derived functional groups in the bioplastic.

Crystallinity of NCC/zein films: The XRD patterns of NCC and NCC/zein bioplastics are shown in Fig. 2. NCC exhibited reflection peaks at $2\theta = 15.78^\circ$ and 23.01° , characteristic of cellulose-I [28–33]. The NCC/zein films showed a broad region between 20° and 23° , which is attributable to the amorphous zein phase [14–21,24–27]. The crystallinity varied among the formulations without a consistent relationship with

TABLE-1
KEY FTIR (cm⁻¹) BAND ASSIGNMENTS OF NCC, ZEIN AND NCC/ZEIN BIOPLASTIC

Sample	C-C	C-O	C-O str.	C-H amine	N-H amide II	C=O amide I	C-H str.	OH str.
NCC	897.90	1031.96	1056.07	1370.48	—	1645.00	2902.99	3416.08
Zein	896.94	1032.93	1078.25	1381.09	1599.06	1652.10	2877.92	3441.16
Bioplastic	895.97	1031.00	1074.40	1307.79	n.d.	1635.71	2879.85	3431.51

n.d. = not detected/defined.

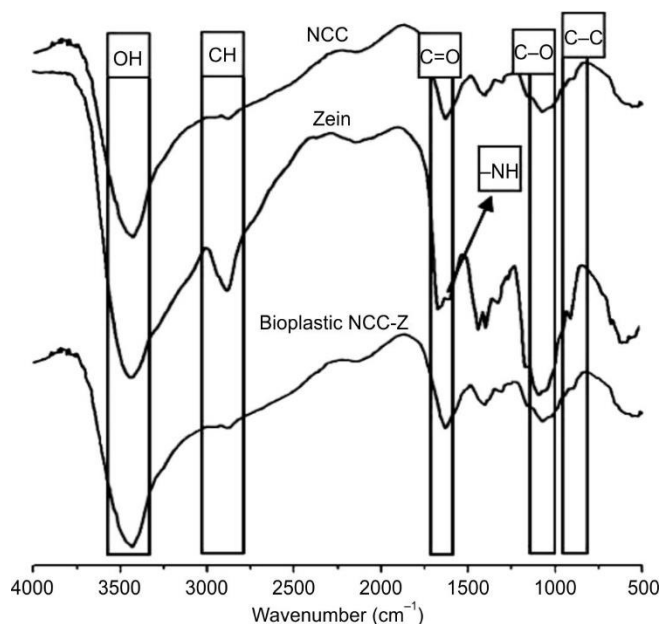


Fig. 1. FTIR spectra of NCC, zein and MAH-coupled NCC/zein bioplastic

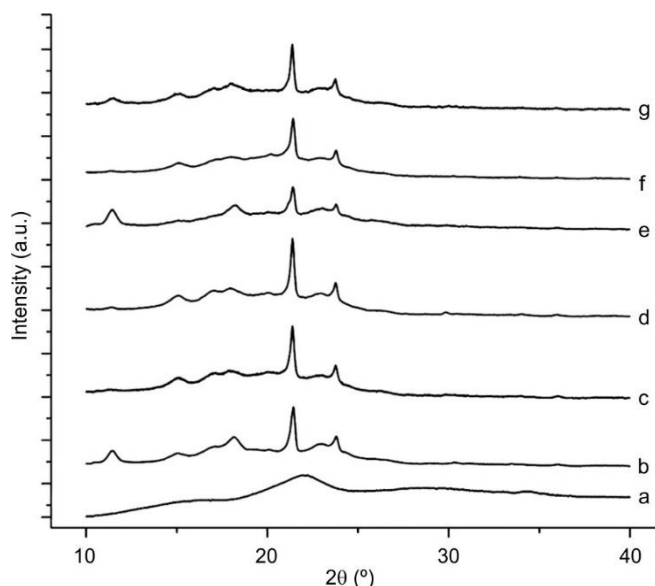


Fig. 2. XRD patterns of NCC and NCC/zein bioplastics: (a) NCC, (b) 0%, (c) 1%, (d) 2%, (e) 3%, (f) 4% and (g) 5% NCC

NCC content (Table-2). Pure NCC showed a crystallinity of 31.00%, while the NCC/zein bioplastics ranged from 18.58% to 30.87%. The highest crystallinity was recorded at 2 wt.% NCC, which may arise from partial matrix organization at low NCC loading, whereas higher NCC contents may promote filler aggregation and phase heterogeneity.

TABLE-2
CRYSTALLINITY AND MECHANICAL PROPERTIES OF MAH-COUPLED NCC/ZEIN BIOPLASTICS

NCC content (% w/w)	Crystallinity (%)	Tensile strength (MPa)	Elongation (%)	Apparent secant modulus at break (MPa)
0	27.083	3.535	10.954	32.27
1	28.784	1.969	3.280	60.02
2	30.874	1.880	4.674	40.22
3	18.579	1.692	3.530	47.93
4	19.495	2.953	6.527	45.24
5	23.529	1.688	3.893	43.35

Mechanical properties: The NCC/zein 0% film showed the highest tensile strength (3.535 MPa) and elongation at break (10.95%). Addition of NCC generally reduced tensile strength and elongation, except for a partial increase in tensile strength and elongation at 4 wt.% NCC (Fig. 3). The trend suggests that the zein-rich film had stronger continuity of the polymer network, while higher NCC loading could induce particle aggregation or local defects that facilitated mechanical failure. Similar competition between reinforcement and aggregation has been reported in biopolymer nanocomposite films [24,25,28,31,34,36].

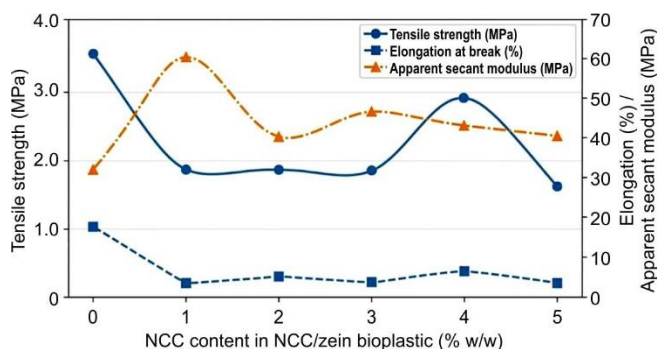


Fig. 3. Tensile response of MAH-coupled NCC/zein bioplastics at different NCC concentrations

The apparent secant modulus at break increased from 32.27 MPa at 0 wt.% NCC to 60.02 MPa at 1 wt.% NCC and then ranged from 40.22 to 47.93 MPa at 2–5 wt.% NCC (Fig. 3). This parameter is reported only as a comparative stiffness index since it is based on ultimate stress and break strain rather than the initial linear slope of the stress-strain curve. Accordingly, the previously reported GPa-scale Young's modulus values have been removed. The corrected tensile-strength range is substantially lower than that reported for highly crystalline or solution-cast NCC/zein systems, emphasizing that processing route, interfacial coupling and filler dispersion strongly influence the mechanical response [24,41,42].

Thermal stability: The thermal decomposition profiles of zein, NCC and NCC/zein bioplastic obtained from TG/DTA are shown in Fig. 4, with the corresponding parameters listed in Table-3. All three materials exhibited two thermal decomposition stages [17,18,33,35]. The first stage was mainly associated with bound-water loss and decomposition of labile groups, while the second corresponded to degradation of the polymeric backbone and char formation. For the bioplastic, the first decomposition stage occurred at 61.9-200.5 °C and the second at 251.2-381.2 °C. The maximum temperature of the second stage was 320.2 °C for the bioplastic, compared with 285.8 °C for zein and 310.3 °C for NCC, which indicates a modest increase in thermal resistance after coupling and blending.

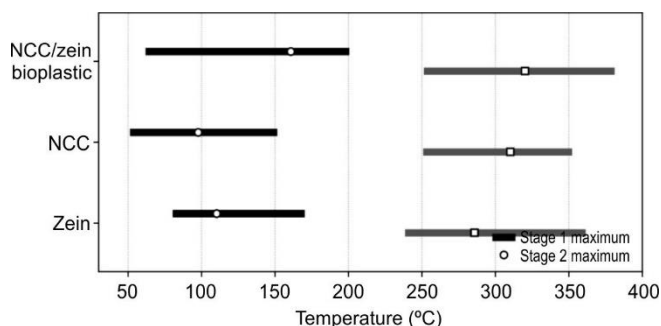


Fig. 4. Thermal decomposition temperature ranges of zein, NCC, and MAH-coupled NCC/zein bioplastic from TG/DTA data

At 490 °C, the bioplastic retained 1.4% residual char, compared with 1.2% for zein and 2.5% for NCC. The absence of a distinct melting transition is consistent with the heterogeneous structure of the NCC/zein matrix. This behaviour agrees with the XRD results, which indicate the coexistence of amorphous zein and partially crystalline cellulose domains [24-33].

Biodegradation and surface morphology: Biodegradation was assessed from mass loss and visible changes in film integrity after 60 days of soil burial in the presence of EM4 microorganisms. The average daily weight-loss rates are given in Fig. 5. NCC/zein 5% exhibited the highest degradation rate ($2.7287\% \text{ day}^{-1}$), followed by NCC/zein 0% ($2.1216\% \text{ day}^{-1}$) and NCC/zein 4% ($2.1039\% \text{ day}^{-1}$). The higher mass loss at 5 wt.% NCC may be related to increased hydrophilicity and greater accessibility of cellulose-rich domains, which can facilitate water uptake and microbial degradation [10-12,36].

SEM images (Fig. 6) of the NCC/zein 0%, 1% and 5% films showed compact and relatively intact surfaces before burial. After degradation, the surfaces exhibited roughness, cracks and irregular features, with the highest damage observed for the NCC/zein 5% film. This surface deterioration agrees with its higher mass-loss rate. NCC content can influence both

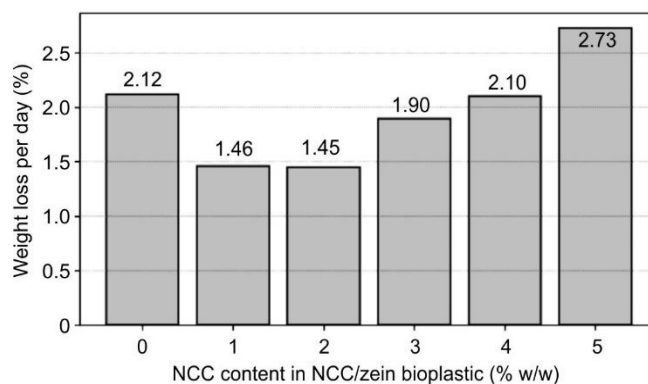


Fig. 5. Average biodegradation rate of MAH-coupled NCC/zein bioplastics during soil burial test

degradation behaviour and mechanical performance, requiring careful control of formulation to balance structural stability during use with degradation after disposal [10-12,36].

Comparison with the related NCC/zein bioplastic systems: Table-4 compares the present films with related NCC/zein systems. Crosslinked NCC/corn-zein films have reported tensile strengths of approximately 64.7-82.6 MPa and Young's moduli in the GPa range, while maleic anhydride-modified CNC-reinforced zein active films have shown tensile strengths of 12.66-37.82 MPa [24,41]. The corrected tensile strength of the present films (1.688-3.535 MPa) is lower than those reported for solution-cast or highly cross-linked systems. Differences in the biomass source, NCC purification, processing route, matrix composition, filler dispersion and coupling efficiency may account for this variation. The present study provides TG/DTA characterization and a 60-day soil-burial assessment across different NCC loadings. Recent NCC/zein systems further indicate that matrix composition, hydrophobicity, wet mechanical behaviour and biodegradability are important considerations in assessing material performance [42,43]. Packaging-related claims remain limited until properties such as barrier performance and surface wettability are directly evaluated.

Conclusion

Maleic anhydride (MAH)-coupled NCC/zein bioplastic films were prepared from sugar palm and maize solid waste through extraction, isolation and melt intercalation. FTIR and ^1H NMR analyses confirmed OH/NH, C-H, C=O and C-O functional groups and supported interactions between NCC and zein within the coupled matrix. XRD analysis showed the coexistence of amorphous zein and partially crystalline cellulose domains, with the highest crystallinity recorded at 2 wt.% NCC. The NCC/zein 0% film exhibited the highest tensile strength and elongation at break, while 1 wt.% NCC provided the highest stiffness. TG/DTA revealed two thermal decom-

TABLE-3
LIST OF KEY THERMAL DEGRADATION PARAMETERS OF ZEIN, NCC, AND NCC/ZEIN BIOPLASTIC

Sample	T1,on (°C)	T1,max (°C)	T1,end (°C)	T2,on (°C)	T2,max (°C)	T2,end (°C)	Temperature at 5% weight loss (°C)	Residual char at 490 °C (%)
Zein	80.5	110.3	170.2	238.4	285.8	361.6	62.2	1.2
NCC	51.2	97.7	151.5	250.8	310.3	352.3	41.0	2.5
NCC/zein bioplastic	61.9	160.8	200.5	251.2	320.2	381.2	54.2	1.4

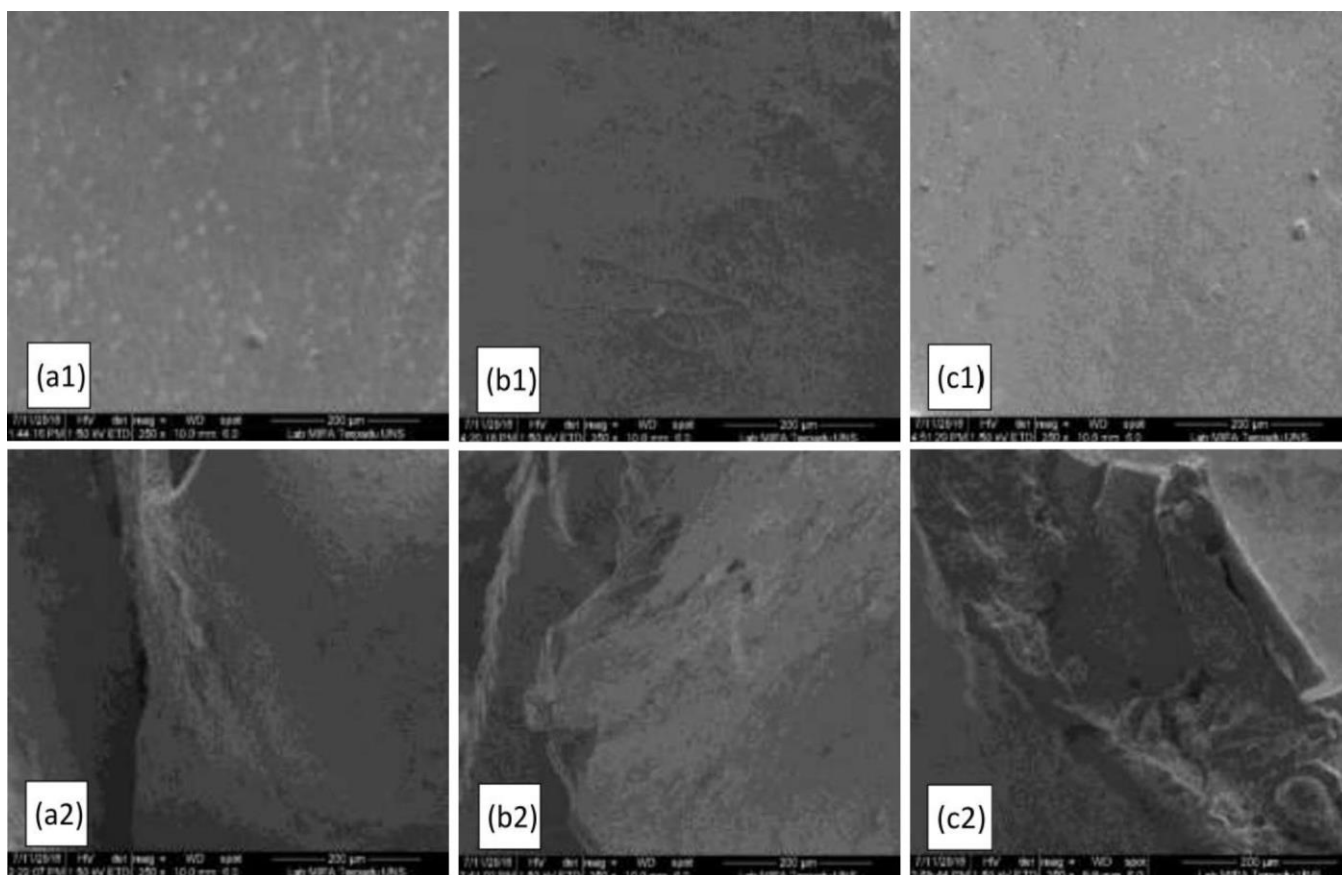


Fig. 6. SEM images of NCC/zein bioplastic films at 250 $\times$ magnification: (a) NCC/zein 0%, (a1) before and (a2) after biodegradation; (b) NCC/zein 1%, (b1) before and (b2) after biodegradation and (c) NCC/zein 5%, (c1) before and (c2) after biodegradation

TABLE-4
COMPARISON WITH RELATED ZEIN/NANOCELLULOSE BIOPLASTIC SYSTEMS

System	Mechanical properties	Thermal/structural features	Biodegradation/barrier information	Ref.
NCC/corn zein + BTCA crosslinking	UTS ~64.7-82.6 MPa; Young's modulus ~3.88-7.73 GPa depending on composition/crosslinking.	Crosslinking and zein altered NCC organization; maximum degradation temperature increased by ~26 $^{\circ}\text{C}$ relative to NCC film.	WVP and OTR decreased at selected NCC/zein ratios; biodegradation not reported in the compared dataset.	[24]
MAH-modified NCC-reinforced zein/catechin/ β -CD nanoparticle film	Tensile strength increased from 12.66 to 37.82 MPa; elongation increased from 4.5 to 5.2% at suitable NCC loading.	FTIR supported intermolecular hydrogen bonding; XRD showed a NCC-related crystalline peak.	WVP, water solubility and swelling decreased; biodegradation not reported in the compared dataset.	[41]
Zein/NCC plant-protein film	Neat zein tensile strength ~9.02 MPa; NCC addition reduced tensile strength in the zein formulation while modifying hardness.	NCC changed film physico-chemical properties; no directly comparable TG/DTA value was reported in the dataset used here.	Water-resistance-related properties were evaluated; no directly comparable soil-burial rate was reported.	[42]
Covalently zein-modified CNF film	Covalent zein modification improved wet mechanical performance.	Covalent attachment of hydrophobic zein to CNF increased hydrophobic character; this is a recent nanocellulose/zein analogue rather than an NCC/MAH system.	Hydrophobicity increased and CNF biodegradability was reported as not being adversely affected.	[43]
MAH-coupled waste-derived NCC/zein film	Tensile strength 1.688-3.535 MPa; elongation 3.280-10.954%; apparent secant modulus 32.27-60.02 MPa.	Crystallinity 18.579-30.874%; T _{2,max} = 320.2 $^{\circ}\text{C}$ for the characterized bioplastic; FTIR/NMR support interfacial interaction.	Soil-burial mass-loss rate 1.4517-2.7287% day ⁻¹ . WVP, oxygen permeability/transmission, water absorption and contact angle were not measured.	Present study

Note: Values are reported as presented in the cited studies. Modulus values are not necessarily directly comparable because test methods and modulus definitions differ among studies.

position stages, with the bioplastic reaching a maximum decomposition temperature of 320.2 °C in the second stage. NCC/zein 5% exhibited the highest biodegradation rate and the highest surface deterioration after soil burial. These findings support the potential of NCC/zein films derived from agricultural biomass as biodegradable materials, while formulation optimization is required to balance mechanical performance with degradation behaviour.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language and no data, results or scientific conclusions were generated by AI. All analyses, interpretations and conclusions are the authors' own. The authors reviewed and edited the content and take full responsibility for the published work.

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