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Biomaterials in Food Packaging

Biomaterials in Food Packaging

**Mohd Yusuf and
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Chapter 7

Characterization of Cassava Starch-Zinc Nanocomposite Film for Food Packaging

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Performance of a pure thermoplastic film can be enhanced by adding nanoparticles of the size ranging 1–100 nm for application in food packaging. This research was carried out to develop a nanocomposite by mechanically homogenizing different proportions of cassava starch (1000 g), glycerol (45–55%, w/w), and zinc nanocomposites (0–2%, w/w). A dispersed mixture of 24 g of these products was then mixed with distilled water (600 ml) and heated for 30 min at boiling temperature to form a thermoplastic solution. The film was formed in different sizes (15, 16, and 17 μm thickness) by casting the solution on a 350 mm $\times$ 180 mm plastic mold of different depths (8, 10, and 12 mm). The performance of the film, including barrier, thermal, and structural properties,

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was determined using standard methods. The results showed that the oxygen and water vapor decreased with the thickness and increased with the concentration of the glycerol. Plastic d-spacing increased with an increase in thickness, and this might indicate the ability of the material to intercalate and exfoliate at some points during a prolonged packaging application. It might also indicate the short-range order of the material constituents in the film for a better service performance. A small degradation of the film was observed between 30 °C and 60 °C. This indicated that the film was thermally stable and might be suitable for packaging applications, especially in the tropical climates. The information of the characterized attributes and optimization of the cassava starch zinc-nanocomposite films justified their alternative application to pure thermoplastic and conventional films for food packaging.

7.1 Introduction

Nanocomposite films are prepared from the homogeneous mixture of renewable raw materials, such as starch, proteins, or any polysaccharide, with a nontoxic plasticizer like glycerol in boiling water at 95 °C [1–4]. Normally, the constituent materials are chosen based on their cost and availability, in order not to interfere with the primary need of the products for food packaging. All kinds of the nanocomposite films, regardless of their source, should have enhanced service performance, and those made from the starch are preferred for economic reason. Due to this, starch is often used in the production of nanocomposite film [3, 4– 6]. However, despite the huge potentials of the starch-based biodegradable materials, the poor service performance in terms of their physical, mechanical, thermal, and structural attributes have greatly limited their applications [7]. Hence, the nanoparticles are often added during the formulation process of the film to provide the attributes required for the packaging applications. The nanoparticles are organic or inorganic materials whose particle size does not exceed 100 nm [8, 9]. These materials can interact with the other material constituents in the biodegradable film because of their large reactive surface area

to volume ratio [5–7]. The resulting blend will thus influence the structural build-up of the molecules, thereby inducing the desired improvement of the film's properties [7].

Many different formulations of nanocomposite films have been reported for application in food packaging [1–7, 9, 10]. For instance, Dang and Yoksan [1] developed a thermoplastic starch from a blend of a plasticized starch and chitosan nanoparticles. Fadeyibi et al. [2, 5, 6] developed a biodegradable film from a blend of the cassava starch and zinc nanoparticles. Chung et al. [3] prepared a biodegradable film from a blend of starch and clay nanocomposites. Dai et al. [7] developed a biodegradable film from a blend of corn starch and taro starch nanoparticles. Tall et al. [9] prepared a biodegradable film from a blend of starch and Ni-Ti nanoparticles. However, the issue with the above-mentioned films is how to solve the possibility of material migration into the packaged food. For this reason, many other types of nanocomposite films are being developed to address this challenge. One way out research has suggested to use a health-friendly nanocomposite material for inclusion in the film formulation. Typically, zinc nanoparticles have been reported to possess this quality and can be used in the formulation of the nanocomposite film, but the quantity added must not exceed 2% (w/w) [2, 5, 6, 10, 11]. Therefore, the study on the characterization of the nanocomposite film prepared from the blend of starch, plasticizer, and zinc nanoparticles cannot be over emphasized.

Application of this class of material in the food packaging has been widely reported. For instance, Fadeyibi et al. [5, 11] used the cassava starch-zinc nanocomposite film to preserve the nutrition qualities, hinder microbial growth, and extend the shelf-lives of tomatoes, cucumber, and garden eggs. Also, Zhang et al. [10] proposed the use of zinc oxide nanoparticles as an effective antimicrobial agent for the use in the preservation of fruits and vegetables. However, the mechanism of this striking behavior of the nanocomposite film has not been reported. Therefore, there is a need to study the characteristics of the nanocomposite film to fully understand its behavior. The objective of this research was to synthesize and characterize the cassava starch-zinc nanocomposite film based on its physical, mechanical, thermal, and structural attributes.

7.2 Materials and Methods

7.2.1 Synthesis and Particle Size Analysis of Nanoparticles

The nanoparticles were synthesized using the sol-gel method described by Fadeyibi et al. [2, 7, 12] and Surya [13] at the processing and storage laboratory, Federal University of Technology, Minna. In this method, a 30 ml of distilled water was added to a solution of 20 ml triethanolamine and 2 ml ethanol, and mixed thoroughly to form a homogeneous solution. This was transferred into a 500 ml beaker containing a 0.5 M solution of 5.39 g zinc acetate di-hydrate and 50 ml distilled water. Another solution of 2 g ammonium hydroxide and 10 ml distilled water was added to the homogeneous mixture in the beaker, which was placed on a Bunsen burner to heat for 20 min. A white bulky precipitate was formed, which was washed 10 times with distilled water before filtration to obtain a clean white residue and dried in an air-circulated oven dryer for 8 h. Moreover, the size distribution of the resulting nanoparticle was determined using a Zetasizer equipment (ver. 7.01) at the Center of Genetic Engineering and Biotechnology, Federal University of Technology Minna, Nigeria. The size of the nanoparticles was found in the range of 4–6 nm.

7.2.2 Development of Nanocomposite Film

The nanocomposite film was developed by mechanically homogenizing different proportions of cassava starch (1000 g), glycerol (45–55%, w/w), and zinc nanocomposites (0–2%, w/w). The choice of the glycerol instead of other types of plasticizers for binding the starch and the nanoparticles together was basically because it did not pose any health hazard when used to prepare materials for food packaging [7, 14]. A dispersed mixture of 24 g of the products was then mixed with distilled water (600 ml) and heated for 30 min at boiling temperature to form a thermoplastic solution. The film was formed in different sizes (15, 16 and 17 μm thickness) by casting the solution on a 350 mm $\times$ 180 mm plastic mold of different depths (8, 10, and 12 mm). The film was thereafter carefully peeled off from the mold and sealed in the polyethylene pack to avoid possible hydration. The process flow diagram for the preparation of cassava starch nanocomposite film is shown in Figure 7.1.

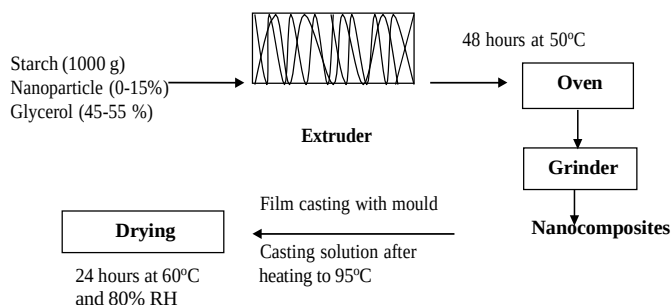


Figure 7.1 Process flow diagram for the development of the nanocomposite film [2, 15].

7.2.3 Determination of Structural and Barrier Properties

A multipurpose X-ray diffractometer was used to study the structural properties, based on the full-width at half maximum and polymer d-spacing of the nanocomposite film, at the Sheda Science and Technology Complex (SHESTCO), Abuja, Nigeria. The range of orderliness of the molecular constituents in the film as well as the degree of intercalation and exfoliation processes in its structure was determined [2]. The ASTM Standard method was used to determine the permeability of the film due to water vapor [16]. This method involves placing an 8.75 cm diameter sample of the nanocomposite film inside a test dish filled with 50 ml deionized water at 100% relative humidity (RH). The setup was subjected to a temperature and RH of 25 °C and 65%, respectively. The test dish was arranged on a rotating turntable to expose it to the same atmospheric conditions for 2 h, before the initial weight of the sample was recorded. The final weight of the sample of the film was obtained after every 15 min, and average values and standard deviations were computed using the expression in Eq. (7.1) [16, 17].

$$W_{\text{vpt}} = \frac{\Delta m}{tA} \quad (7.1)$$

where, Δm is change in mass (g), t is the time (min), A is the area of the mouth of the test dish (m^2), and W_{vpt} = water vapor transmission rate ($\text{g}^{-2}\text{s}^{-1}$).

Thus, the water vapor permeability of each film sample was computed using Eq. (7.2) [16].

$$W_{vp} = \frac{W_{vpt}}{\Delta p} \times T \quad (7.2)$$

where T is the thickness of the film sample (mm), Δp the partial vapor pressure difference across the film, and W_{vp} the water vapor permeability (g/m.Pa.s).

Furthermore, the method described by Fadeyibi et al. [2] was used to determine the oxygen gas permeability of the nanocomposite film. In this method, 2 g of the film sample was placed inside an absorption column setup, which was stocked at one end, and stream of oxygen gas was passed into the column via the open end at the top [17, 18]. The concentration of the oxygen gas inside the column was measured using an electronic weighing balance for 1 h at an interval of 10 min. Thus, knowing the data of properties given in Table 7.1, the average value and standard deviation of the oxygen permeability of each sample of the nanocomposite film were computed using Eqs. (7.3–7.8).

Table 7.1 Data of properties used for the calculation of oxygen permeability

	Variable	Symbol	Value	SI unit
1	Volume of column	V_v	0.0174	cm ³
2	Density of oxygen	P_{O_2}	0.001331	g/m ³
3	Atmospheric pressure	P_{atm}	101325	Pa
4	Saturated water vapor pressure	$P_{H_2O}^{sat}$	$190.2 \times T$	°C Pa
5	Area of film sample	A	0.000123	m ²
6	Atmospheric temperature	T	25	°C

$$[O_2] = \frac{mo_2}{1000M} \quad (7.3)$$

$$[O_2]_n = \frac{RH \times P_{H_2O}^{sat}}{100RT} \quad (7.4)$$

$$[O_2] \% = \frac{O_2}{[O_2]_n} \times 100 \quad (7.5)$$

Therefore,

$$P_t = P_{atm} - P_{H_2O}^{sat} \times T \quad (7.6)$$

$$\Delta PO_2 = ([O_2]_t - [O_2]_n)P_t \quad (7.7)$$

$$O_p = \frac{\Delta m \Delta y}{A \Delta t \Delta PO_2} \quad (7.8)$$

where $[O_2]_t$ is the oxygen concentration inside the absorption column at time t , $[O_2]_n$ the initial column oxygen content, the mass of oxygen gas absorbed by the cassava starch nanocomposite film in the column, M the molar mass of oxygen gas (16 g/mol), R the molar gas constant ($J/mol \cdot K$), RH relative humidity (63%), T absolute temperature (25 + 273 K), O_p oxygen permeability (g/m.Pa.s), and Δy the thickness of the film (μm).

7.2.4 Determination of Thermal and Rheological Properties

A thermogravimetric analyzer was used to determine the thermal properties of the nanocomposite film. The weight degradation or loss of the film was determined by computing its difference in weight after exposure to a laboratory-controlled temperature 30–950 °C for 30 min. The rheological properties of the nanocomposite films were determined using a nano-mechanical testing machine (Model: Hysitron TI750) at the SHESTCO, Abuja, Nigeria. A record of the applied load on the film sample, depth of penetration of an indenter, and the corresponding indented area were determined by the machine's response. The values of the young modulus, hardness, activation volume, and tensile strength of the sample of the nanocomposite film were thereafter computed from a graph of the load versus the penetration depth or displacement (Figure 7.2.), and using the empirical relationships in Eqs. (7.9–7.14) [19].

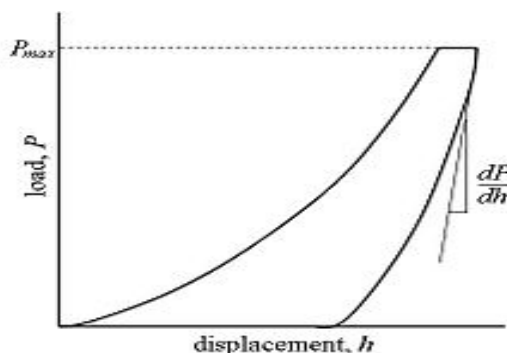


Figure 7.2 Load-displacement curve of a sample of nanocomposite film [19].

$$E = dP/dh \quad (7.9)$$

$$Y_r = \frac{S_2}{2\alpha} \times \sqrt{\frac{\pi}{A_i(h_a)}} \quad (7.10)$$

$$H = \frac{P_{\max}}{A_p} \quad (7.11)$$

$$m = \frac{d \ln \sigma}{d \ln \dot{\delta}} \quad (7.12)$$

$$\dot{\nu} = \frac{9k_b T \ln \dot{\delta}}{dH} \quad (7.13)$$

$$T_s = \frac{P_{\max}}{A_c} \quad (7.14)$$

where dP/dh is the slope of the load displacement curve (N/m), S stiffness of the contact of the indenter with film sample, α geometric constant of the material ($= 1$), h_a contact depth (μm), A_p projected area of the indenter (μm^2), Y_r the reduced modulus of the film sample ($\mu\text{N}/\text{mm}^2$), H hardness of the film sample (MPa), $P_{\max}$ the maximum applied load (μN), σ flow stress of the film due to indenter penetration ($\mu\text{N}/\text{mm}^2$), strain or drift rate of the film under indentation (nms^{-1}), m stress strain sensitivity of the flow ($\mu\text{N}/\text{mm}^2$), k_b Stephen Boltzmann's constant, T temperature of the film sample ($^{\circ}\text{C}$), activation energy (kJ), A_c cross-sectional area of the film sample (mm^2), and T_s tensile strength of the film sample (N/mm^2).

7.3 Results and Discussion

7.3.1 Permeability of Nanocomposite Film

The study of water vapor permeability of food packaging materials is important for maintaining or extending the shelf life of the food product. The water vapor permeability coefficient indicates the amount of water vapor that permeates per unit of area and time in the nanocomposite film. The results of the permeability of the various categories of the nanocomposite film after 3 h exposure of water and oxygen gas are presented in Table 7.2. The variation of the water vapor and oxygen permeability with the concentration glycerol

and the level of zinc nanoparticles are affected by the thickness of the film. The amount of water vapor, absorbed by the film, decreases with the concentration of the zinc nanoparticles and increases with the concentration of the glycerol. The molecular absorption behavior of the nanocomposite films may be influenced by the nanoparticles used. The water vapor permeability is low for the film without the nanoparticles and high for the film with 2% nanoparticles. The reason for this may be associated with the higher water absorption affinity of nanoparticles at higher concentrations. The addition of the nanoparticles to the film may have caused a modification of the polymer network of the film, thereby affecting its water vapor permeability. An increase in the glycerol concentration from 45% to 55% may even cause an increase in the mobility of the polymeric chains of the film [20]. The overall effect of the composition of the film on the water vapor permeability of the material can be enhanced with the addition of more nanoparticles and glycerol. The addition of more glycerol may decrease the intermolecular hydrogen bonds between the polymer chains, thereby increasing the flexibility of the nanocomposite film [20, 21]. The nanocomposite film is, therefore, an ideal material for packaging low-moisture food products because of its high-water vapor permeability coefficient.

The knowledge of oxygen gas permeability of the nanocomposite film is essential for understanding the rate of product respiration and shelf life. The amount of oxygen gas absorbed by the film decreases with the increase in concentrations of zinc nanoparticles and glycerol (Table 7.2). The oxygen permeability is generally higher for the film without the nanoparticles, and this decreases with the increase in level of nanoparticles and glycerol. The presence of the nanoparticles in the film is likely to alter the gas mobility, as they diffuse across the film. Although the reason for this behavior is not yet known, it is likely that the decrease in the porosity of the nanocomposite film as a result of an increase in amount of the nanoparticles may be responsible for this observation. It may also be related with the formation of a stable complex resulting from the filling up of the empty d-spaces of the zinc nanoparticles with the oxygen gas molecules [2]. Therefore, the rate of diffusion of the oxygen gas across the film layers may reduce due to the decrease in number of pore space or pinhole opening of the film. Thus, the nanocomposite film can help reduce the amount of atmospheric

Table 7.2 Permeability of cassava starch nanocomposite films for 3 h period

Permeability of cassava starch nanocomposite films (g/m.Pa.s)								
Film of 15 μm			Film of 16 μm			Film of 17 μm		
Treatments	WVP [$\times 10^{-10}$]	OP [$\times 10^{-11}$]	Treatments	WVP [$\times 10^{-10}$]	OP [$\times 10^{-11}$]	Treatments	WVP [$\times 10^{-10}$]	OP [$\times 10^{-11}$]
45-0-15	2.240 $\pm$ 1.076	1.568 $\pm$ 0.084	45-0-16	1.723 $\pm$ 0.101	1.521 $\pm$ 0.018	45-0-17	0.613 $\pm$ 0.023	1.240 $\pm$ 0.002
45-1-15	2.360 $\pm$ 0.049	1.530 $\pm$ 0.016	45-11-6	1.730 $\pm$ 0.119	1.500 $\pm$ 0.017	45-1-17	0.797 $\pm$ 0.006	1.109 $\pm$ 0.082
45-2-15	2.450 $\pm$ 1.027	1.437 $\pm$ 0.030	45-2-16	1.837 $\pm$ 0.112	1.287 $\pm$ 0.081	45-2-17	0.813 $\pm$ 0.053	1.192 $\pm$ 0.008
50-0-15	3.170 $\pm$ 0.001	1.347 $\pm$ 0.041	50-0-16	1.840 $\pm$ 0.008	1.169 $\pm$ 0.009	50-0-17	1.221 $\pm$ 0.003	1.151 $\pm$ 0.043
50-1-15	3.380 $\pm$ 0.001	1.320 $\pm$ 0.087	50-1-16	1.873 $\pm$ 0.001	0.886 $\pm$ 0.004	50-1-17	1.323 $\pm$ 0.002	0.633 $\pm$ 0.058
50-2-15	3.420 $\pm$ 0.000	1.280 $\pm$ 0.011	50-2-16	1.874 $\pm$ 0.002	0.789 $\pm$ 0.005	50-2-17	1.340 $\pm$ 0.001	0.614 $\pm$ 0.063
55-0-15	3.450 $\pm$ 0.000	1.184 $\pm$ 0.020	55-0-16	2.032 $\pm$ 0.001	0.657 $\pm$ 0.004	55-0-17	1.424 $\pm$ 0.003	0.502 $\pm$ 0.004
55-1-15	3.640 $\pm$ 0.001	0.917 $\pm$ 0.050	55-1-16	2.040 $\pm$ 0.001	0.589 $\pm$ 0.017	55-1-17	1.428 $\pm$ 0.004	0.486 $\pm$ 0.014
55-2-15	3.680 $\pm$ 0.003	0.807 $\pm$ 0.075	55-2-16	2.059 $\pm$ 0.002	0.481 $\pm$ 0.006	55-2-17	1.513 $\pm$ 0.001	0.456 $\pm$ 0.023

oxygen gas influx into the system of the packaged food product. This may create a macro environment or modified atmosphere for the ideal storage or packaging of the food product.

Thickness is structurally related with the permeability of the nanocomposite film [22, 23]. The thickness of the film plays an important role in its absorption behavioral and characterization. Thus, the variations in barrier property of the nanocomposite film with an increase in its thickness are shown in Table 7.2. There is a clear decrease in the water vapor and the oxygen gas permeability from 2.240×10^{-10} to 0.613×10^{-10} g/m.Pa.s, with the increase in the thickness of the film. The permeability of the film is slightly higher as compared with the permeability of low- and high-density polyethene materials of similar thickness [23]. In a related investigation, Islam and Buschatz [24] observed an increase in the permeability of polyamide films with the increase in their thickness. Thus, the thinner the nanocomposite film, the higher will be the diffusion and water vapor coefficients, due to the less restriction of particle influx into the film layers.

7.3.2 Thermal Stability of Nanocomposite Film

A typical thermal degradation curve for different thicknesses of the cassava starch nanocomposite film, which were obtained for the film with 45% glycerol and 0% zinc nanoparticles, is shown in Figure 7.3. Similar trends were observed for the remaining 26 film formulations. Less than 2% of the weight of the film was degraded between 30 °C and 85 °C. The residual weights of the film with 45% glycerol and 15–17 µm thickness were 14%, 12%, and 9% at 30 °C.

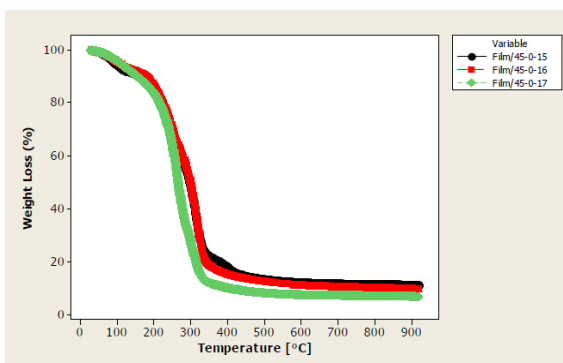


Figure 7.3 Thermal degradation curves of nanocomposite film.

The residual weights of the films with 45% glycerol and 1% zinc nanoparticles were 13% for 15 μm , 10% for 16 μm , and 12% for 17 μm . The films containing 45% glycerol and 2% zinc nanoparticles offered 17%, 16%, and 10% residual weight loss with the increase in thickness. Also, less than 2% of the weights of the films were degraded between 30 °C and 70 °C. Furthermore, only 2% of the weights of the cassava starch nanocomposite films were degraded between 30 °C and 50 °C, in the case of the films with 50% glycerol and 1% zinc nanoparticles. In general, since more than 80% of the films were degraded when subjected to temperature above their glass transition range, the residual weight represented almost complete decompositions of the films.

Beyond this temperature range, the cassava starch nanocomposite film may become unstable and the weight loss of the film may increase with the increase in temperature. The differences in the values of the glass transition temperatures of the films and their residual weights can be partly associated with their varied thicknesses, which affect the physical states, volumes, and shapes of the samples at higher temperature. It can also be argued that the increased concentrations of the glycerol and the zinc nanoparticles are partly responsible the difference in the glass transition temperature. This behavior agrees with the low-density polyethylene film and other synthetic polymers, which show a single mass loss step with significant decomposition [25]. Therefore, the knowledge of thermal properties of the nanocomposite film is necessary for understanding its decomposition pattern for packaging application, especially in a high temperature environment.

7.3.3 Mechanical Properties of Nanocomposite Film

The mechanical properties of the nanocomposite film may be necessary for studying the influence of defects such as pinholes that may affect other properties, such as the resistance to breakage and abrasion. The effects of nanoparticles and glycerol concentrations on the mechanical properties of the nanocomposite film are shown in Table 7.3. The reduced modulus and the strain energy decrease with the increase in the nanoparticles and glycerol concentrations. Also, the hardness, drift rate, tensile strength, and deformation increase with the increase in the concentrations of glycerol and

zinc nanoparticles. Although the reason for this is not well known, from previous research and preliminary investigation [26–30], the morphology of the film and its displacement when load is applied are evaluated. This may create a large interface between the film constituents', thereby affecting its molecular mobility and relaxation behavior. This corroborates the findings of Park et al. [31], who reported that the polymer structure, plasticizer selection and concentration, molecular weight of the film forming materials, type of solvents, and film thickness affect the mechanical properties.

Furthermore, the results of the effects of thickness on the loading and unloading behaviors of cassava starch nanocomposite films are typically shown in Figure 7.4. The variations of the applied load on the cassava starch nanocomposite films with the displacement, observed in the nano-indentation analyses, are also shown in Figure 7.4. The strain energy absorbed by the cassava starch nanocomposite film is higher for the 15 μm film than for the 16 μm and 17 μm , thus a magnitude of 190 pNm is released during the loading part of the hysteresis loop. But, only 5 pNm of the strain energy is dissipated as heat during the unloading part of the hysteresis loop, as most of the energy is recovered in the process. It is possible that the energy dissipated as heat during the unloading part of the loop by the cassava starch nanocomposite films is lower than those of the pure thermoplastic films, irrespective of the concentration of the glycerol [32]. The energy dissipated also decreases with the thickness of the films.

Table 7.3 Mechanical properties of cassava starch nanocomposite films

Variables			Mechanical properties of the films						
<i>g</i>	<i>np</i>	<i>t</i>	<i>E</i>	<i>H</i>	φ	<i>d</i>	σ_t	δ	<i>F</i>
45	0	15	1.39	941.1	101.0	11.34	0.400	4040.2	50.00
45	1	15	2.47	765.6	80.80	3.862	0.000	3231.6	50.00
45	2	15	3.19	515.3	74.28	−7.63	0.000	2977.0	49.90
45	0	16	5.39	1.010	55.91	−1.57	0.000	2236.5	50.00
45	1	16	43.7	2.690	24.95	12.64	0.200	998.10	50.00
45	2	16	7.01	701.6	55.02	8.478	0.100	2200.7	50.00
45	0	17	2.76	163.7	103.3	−0.76	0.100	4132.8	50.00
45	1	17	6.34	14.11	155.4	−2.31	0.100	6214.7	50.00

(Continued)

Table 7.3 (Continued)

Variables			Mechanical properties of the films							
g	np	t	E	H	ϕ	d	σ_t	δ	F	
45	2	17	10.2	1.480	42.01	-3.60	0.100	1680.5	50.00	
50	0	15	4.73	433.9	68.75	5.46	0.100	2749.9	50.00	
50	1	15	5.09	311.3	33.11	-3.05	0.200	1324.4	50.00	
50	2	15	5.62	286.5	75.94	-0.17	0.100	3037.6	50.00	
50	0	16	5.52	1.110	54.82	11.72	0.000	2192.8	50.00	
50	1	16	92.4	73.50	14.54	-1.54	0.100	581.50	50.00	
50	2	16	11.3	1.090	43.49	6.90	0.100	1739.5	50.00	
50	0	17	17.2	1.410	36.78	24.3	0.100	1471.2	50.00	
50	1	17	12.2	391.3	61.28	-3.84	0.200	2451.0	50.00	
50	2	17	13.5	4.040	34.22	25.4	0.100	1368.8	50.00	
55	0	15	168	67.86	9.556	-0.28	0.200	383.00	49.90	
55	1	15	60.7	5.600	18.67	-1.41	0.200	746.80	50.00	
55	2	15	45.2	4.980	20.8180	-10.1	0.100	832.70	50.00	
55	0	16	41.3	33.87	22.0480	-0.70	0.100	881.90	50.00	
55	1	16	167	67.86	9.55590	-0.28	0.200	383.00	49.90	
55	2	16	12.9	7.060	542.909	-7.47	0.1	3620.6	299.9	
55	0	17	43.40	35.51	21.5030	-2.56	0.1	860.10	50.00	
55	1	17	185.2	11.43	11878	4.88	0.4	475.10	50.00	
55	2	17	84.32	9.070	15170	-1.55	0.2	606.80	50.00	

Note: = Drift rate (nm/s), = Maximum force (μN), = Maximum depth (nm), = Tensile strength ($\mu\text{N}/\text{mm}^2$), = Hardness (MPa), = Reduced modulus ($\mu\text{N}/\text{mm}^2$), = Strain energy (pNm), g = Glycerol concentration (% w/w), np = Concentration of nanoparticles (% w/w), t = Thickness of the film (μm).

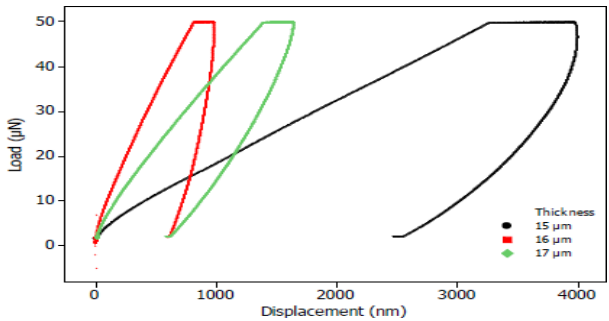


Figure 7.4 Load-displacement profiles of nanocomposite film [29].

7.3.4 Structural Stability of Nanocomposite Film

The results of some structural properties, showing the X-ray diffraction patterns of the cassava starch nanocomposite films of different thicknesses and concentrations of glycerol and nanoparticles, are typically shown in Figure 7.5. The peak values of the structural properties at 15 μm , 16 μm , and 17 μm film thicknesses, which were deduced from the X-ray diffraction patterns, are presented in Table 7.4.

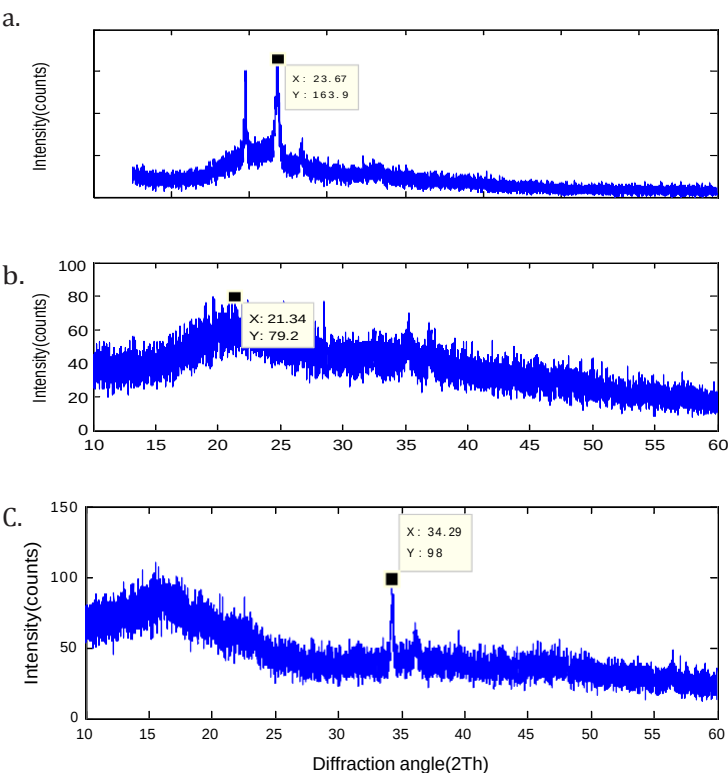


Figure 7.5 Profile of nanocomposite film with 2% nanoparticles: (a) 45% glycerol, (b) 50% glycerol, and (c) 55% glycerol.

Table 7.4 Peak values of cassava starch nanocomposite films

Table 7.4 (Continued)

Structural properties of cassava starch Nanocomposite films							
Variables			Position	Height	FWHM	d-spacing	
<i>g</i>	<i>np</i>	<i>t</i>	[°2Th]	[cts]	[°2Th]	[Å]	[%]
45	0	15	10.12	10.79	0.19	8.74	65.22
45	1	15	5.27	6.71	0.5	16.77	7.7
45	2	15	35.06	12.54	0.76	2.56	65.74
45	0	16	10.12	10.79	0.19	8.74	65.22
45	1	16	10.12	10.79	0.19	8.74	65.22
45	2	16	23.19	34.56	0.25	3.84	48.35
45	0	17	5.27	6.71	0.5	16.8	7.7
45	1	17	34.39	21.02	0.25	2.61	100
45	2	17	23.29	15.96	0.76	3.82	75.92
50	0	15	35.12	11.62	0.19	2.56	100
50	1	15	23.33	27.86	0.5	3.81	67.68
50	2	15	22.87	20.09	0.63	3.89	100
50	0	16	22.87	20.09	0.63	3.89	100
50	1	16	5.27	6.71	0.5	16.8	7.7
50	2	16	24.63	8.3	0.92	3.61	100
50	0	17	34.54	17.44	0.19	2.6	100
50	1	17	23.88	13.64	0.61	3.72	100
50	2	17	17.38	9.05	0.76	5.1	15.34
55	0	15	34.54	17.44	0.19	2.6	100
55	1	15	17.23	10.39	0.76	5.15	27.55
55	2	15	10.48	10.82	0.5	8.44	65.4
55	0	16	19.56	104.3	0.16	4.54	100
55	1	16	35.24	15.62	0.5	2.55	100
55	2	16	18.62	41.17	0.88	4.76	100
55	0	17	34.26	43.48	0.16	2.62	100
55	1	17	32.16	17.64	0.5	2.78	71.76
55	2	17	34.42	7.21	0.38	2.61	68.25

Note: FWHM, full width at half maximum; *g*, glycerol concentration (% w/w); *np*, concentration of nanoparticles (% w/w); *t*, thickness of the film (μm).

The X-ray diffraction is a commonly used technique for the evaluation of crystal structure because it relies on coherent scattering from many unit cells in a spatially time averaged fashion [33]. The effects of the intensity of incident radiation on the pure thermoplastic films of different thicknesses, containing 45% glycerol, are shown in Table 7.4. The diffraction patterns depicted show a defect in the 15 μm thermoplastic film, which occurs at a diffraction angle of $19.68\ 2\theta^\circ$, unlike the other thermoplastic films that show no observable defect with diffraction. The effects of the intensity of incident radiation on the cassava starch nanocomposite films of different thicknesses, containing 45% glycerol and 1% zinc nanoparticles. The diffraction patterns depicted show defects in the 15 μm and 16 μm cassava starch nanocomposite films, which occur at diffraction angles of $19.62\ 2\theta^\circ$ and $19.68\ 2\theta^\circ$, respectively; unlike the 17 μm film that shows no observable defect with diffraction. The effects of the intensity of incident radiation on the cassava starch nanocomposite films of different thicknesses, containing 45% glycerol and 2% zinc nanoparticles, are also shown in Table 7.4. The diffraction patterns depicted show defects in the 15 μm and 17 μm cassava starch nanocomposite films, which occur at diffraction angles of $23.2\ 2\theta^\circ$ and $19.44\ 2\theta^\circ$, respectively; unlike the 16 μm film that shows no observable defect with diffraction. It can be seen, from the foregoing, that thickness is important in determining the crystalline structures of cassava starch nanocomposite films, in terms of the diffraction position along the films' geometry. The observed patterns for the variations of the intensity with the diffraction angles are relatively homogeneous. The patterns show relatively sharp peaks, which indicate that the nanocomposite film may be crystalline in its structure. This resembles the structure of a face-centered cubic zinc particle with a lattice constant of approximately 0.4085 nm and is consistent with the available literature value (0.4086 nm) [2, 27]. The film-forming materials and especially their structural cohesion have important effects on the structural properties of edible films and coatings [27]. In addition, an area of almost 100% structural defect intensity in the molecular structure of the nanocomposite film is found, as shown in Table 7.4. The intensity of the structure of the film depends on the material composition, as evident in the different

patterns exhibited by the various film categories and treatments. The defect is more pronounced in the film with 55% glycerol than the film with either 50% or 45% glycerol concentration. Also, the values of the full width at half maximum can help further explain the size of the observed crystals in the film. An understanding of this phenomenon may be vital for the food packaging engineer in the designing of systems for the food packaging application. It may also aid in the understanding of barrier mechanism of nanocomposite film, which is yet to be reported.

7.4 Conclusion

The service performance analysis of the cassava starch-based zinc nanocomposite film is carried out to ascertain its suitability for packaging application. This is done by characterizing the film based on its mechanical, thermal, structural, and barrier properties. The results show that the amount of water vapor absorbed by the film decreases with the increase in concentrations of the zinc nanoparticles and the glycerol. The molecular absorption behavior of the nanocomposite films may be influenced by the nanoparticles used. The water vapor permeability is low for the film without the nanoparticles and high for the film with 2% nanoparticles. The residual weights of the films with 45% glycerol and 1% zinc nanoparticles are 13% for 15 μm , 10% for 16 μm , and 12% for 17 μm . The films containing 45% glycerol and 2% zinc nanoparticles offer 17%, 16%, and 10% residual weight loss with the increase in their thickness. The reduced modulus and the strain energy decrease with the increase in the nanoparticles and glycerol concentrations. The hardness, drift rate, tensile strength, and deformation increase with the rise in concentrations of glycerol and zinc nanoparticles. The diffraction patterns depicted show a defect in the 15 μm thermoplastic film, which occurs at a diffraction angle of 19.68° , unlike the other thermoplastic films, which show no observable defect with diffraction. Thus, the nanocomposite film has a potential for use as an active packaging material in the food processing.

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