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Nano-Rheological Behaviour of Cassava Starch-Zinc Nanocomposite Film under Dynamic Loading for High Speed Transportation of Packaged Food

Adeshina Fadeyibi, Zinash D. Osunde, Gbabo Agidi,
Evans C. Egwim and Peter A. Idah

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Abstract

This research was undertaken to determine the nano-rheological behaviours of cassava starch-zinc-nanocomposite films under dynamic loading for assessing their suitability as food packaging materials in high speed transportation. The films, with thickness ranging between 15 ± 0.22 – 17 ± 0.13 μm , were prepared by casting mixtures of 24 g cassava starch, 45–55% (w/w) glycerol and 0–2% (w/w) zinc nanoparticles in plastic moulds of 8–12 mm depths. The effects of the nanoparticles, thickness and glycerol on the rheological properties of the films, including the Young's modulus, creep, hardness and plasticity index were determined using nanoindentation technique. The results show that the Young's modulus and hardness of the films varied inconsistently with glycerol concentration and nanoparticles due probably to their isotropic nature and sensitivity to slight change in load. The plasticity index was lower for 15 μm film, which absorbed 40 pNm and dissipated 0.5 pNm during loading and unloading stages, respectively. The response of the 15 μm film to creep was higher than 16 μm and 17 μm films, and this may be consequence of lower wear at higher loads. This implies that the nanocomposite film might be suitable for high speed transportation of packaged food.

Keywords: rheological behaviour, hysteresis loop, zinc nanocomposite film, food packaging

1. Introduction

Flexible films (<100 μm), such as low-density polyethylene and polyvinyl chloride, are widely applied in the food industry [1]. The handling of the flexible films in the packaging and

transportation of food and food products over long distances is an issue bordering the agricultural products processing engineers, due to the dynamic loading experienced by the materials in the process [2]. The viscoelastic behaviour often exhibited by this material has great influence on its degree of wear and tear, especially during high speed transportation of packaged foods. The low mechanical impedance of the flexible films has rendered measurement of the rheological behaviour difficult to achieve using the universal tensile testing equipment but can be measured using nanoindentation analysis [3, 4]. The indirect measurement of the area of contact between the indenter and flexible film under investigation is called nanoindentation. The method provides an avenue of touching the flexible film, whose rheological properties are unknown, with another material of known property. The penetration depth of the indenter, which allowed for proper measurement of the penetration rate in the flexible film, is usually measured in nanometres, thus considering the process as non-destructive because of its relatively smaller area of contact. The accurate measurement of the rheological properties has been made possible using the nanoindentation technique. It is easier to measure the rheological properties of flexible films by this process due probably to the high response of the material to the depth sensing device. The knowledge of the response of flexible films to this device can be used to study their behaviour with respect to deformation or wear [5].

The rheological behaviour of flexible films, under static and dynamic loading, has been reported by some researchers [6–8]. Jian et al. [6] studied the load-displacement behaviour of Cu_2O thin films by nanoindentation. The authors observed that the regions of loading and unloading appeared distinct on the hysteresis loop of the films. Also, Syed et al. [7] studied the nanoindentation behaviour of ultra-thin polymeric films and use the finite element modelling to characterise their mechanical properties. The authors observed that the Young's modulus and hardness of the flexible film increased with the indentation depth and the projected area of the deformed region. The nano-rheological properties of flexible films including the hardness and elastic modulus have been studied by Chateauinois and Briscoeb [8]. The authors observed a progressive wearing of the flexible film due to compaction brought about by increasing load at the contact area. The intensity of the wearing process was interpreted by considering the evolving load carrying capacity of the contact, which was characterised by progressive redistribution of the contact pressure within the flexible film. However, the behaviour of the flexible films, as reported, does not account for the viscoelasticity, elastic and plastic works of the materials. It was also difficult to determine the plasticity index and creep of the materials because of their inconsistent strain rate sensitivity under dynamic loading. The hardness and Young's modulus of the materials were practically inadequate to withstand the dynamic vibrational loading usually experienced on rough roads. Many of the flexible films therefore are limited in their application for high speed transportation, where higher hardness, Young's modulus and elastic behaviours of the materials are required for food packaging. Hence, there is the need for a suitable food packaging material with the required characteristics for potential application in high speed transportation of packaged foods.

The addition of zinc nanoparticles to renewable resource like starch provides an alternative flexible film with improved rheological properties. The new material offers opportunity to

package food under dynamic loading at high speed due to their improved rheological behaviour. The improvement of these properties may be explained by the fundamental length scales of the nanoparticles, whose uniform dispersion with the starch results in ultra-large interfacial area between the constituents. The interface between the organic and inorganic materials alters the mobility of the molecules and thus the rheological behaviour of the nanocomposite material. Thus, the objective of this research was to determine the nano-rheological behaviour of cassava starch-zinc nanocomposite film, such as hardness, Young's modulus, creep, elastic and plastic works, under dynamic loading for high speed transportation of packaged food.

2. Materials and methods

2.1. Source of materials

The cassava used in this investigation was bought from Kasuwan Gwari Market in Minna, Niger State. The method described by Fadeyibi et al. [9] was used to prepare the starch slurry, which was dried under the Sun for 1 week until a moisture content of 2 % (wb) was achieved.

2.2. Preparation and size analysis of zinc nanoparticles

The method described by Fadeyibi et al. [10] was used to prepare the zinc nanoparticles. The method involves mixing two homogeneous solutions prepared separately. The first solution was prepared by adding 30 mL of distilled water to 20 mL of triethanolamine with constant stirring while adding 2 mL (100 drops) of ethanol in drop wise. The second solution was prepared by adding 5.39 g of zinc acetate di-hydrate to 50 mL distilled water with continuous stirring, to form a 0.5 M solution. The two solutions were mixed together in 500 mL beaker and a solution of ammonium hydroxide and 10 mL distilled water were added (in drop wise). The mixture was left undisturbed for 30 minutes to form a white bulky solution, which was washed 8–10 times with distilled water and filtered in a filter paper. The resulting residue was dried in the oven at 95°C for 8 hours. Also, the zetasizer equipment (version 7.01) was used to carry out size analysis of the synthesised zinc particles to establish their size range in nanometre. The tests were carried out at the Centre of Genetic Engineering and Biotechnology of the Federal University of Technology Minna, Nigeria.

2.3. Preparation of cassava starch-zinc nanocomposite film and thickness determination

The starch composite was prepared by adding analytical grade glycerol (45–55%, w/w) and zinc nanoparticles (0–2%, w/w) to 24 g of the prepared cassava starch. The mixtures were homogenised using a locally fabricated screw extruder to form the nanocomposites. The resulting nanocomposites were dispersed into 600 mL of distilled water to form suspensions and heated for 30 minutes until viscous thermoplastic liquid were formed. The thickness of the film was determined by casting the thermoplastic solution in plastic mould of 8, 10 and 12 mm depths. In the design of the plastic mould, the surface area was first determined by

wrapping 50 g of biomaterials (50 mm major and 10 mm minor axial dimensions) using aluminium foil and the layout was traced on the graph paper. The size of the plastic mould was computed from the empirical relationship expressed in Eq. (1).

$$TSA = Na + e \quad (1)$$

where N is the number of equivalent biomaterials (50 g), a is the surface area of the mould mm^2 , e is the allowance (assumed 600 mm^2) and TSA is the total surface area of the mould (mm^2).

Based on the expression in Eq. (1), the total surface area of the mould measured was $350 \times 180 \text{ mm}$ and this was used to cast the thermoplastic solutions into films. The thickness of the dried film, whose moisture content was 4% (db), was determined at the four edges of the films and the average taken. The plastic mould with 8 mm depth gave an average dried thickness of $15.14 \pm 0.22 \mu\text{m}$, whereas those with 10 and 12 mm depths were 16.21 ± 0.36 and $17.38 \pm 0.13 \mu\text{m}$, respectively. Twenty-seven samples of the cassava starch-zinc nanocomposite film, obtained from the 3^3 full factorial experiments (three levels from each of the zinc nanoparticles, glycerol and thickness), were prepared and stored in separate polyethylene bags to avoid subsequent hydration.

2.4. Determination of rheological properties of the film

The nanoindenter was used to determine the rheological properties of the nanocomposite films. A typical profile of the load-displacement curve of the film, obtained from the nanoindenter, is shown in **Figure 1**. The profile shows loading, unloading and holding stages from which other rheological behaviours such as the hardness, Young's modulus and creep were computed (Eqs. 2 and 3). Also, the strain rate sensitivity, which corresponds to creep response of the films, was determined at the holding stage of the profile (stage 2). The elastic and plastic works correspond to the areas under the loading and the unloading stages of the hysteresis loop [7, 11]:

$$H = \frac{P_{max}}{A_c h_c} \quad (2)$$

where P_{max} is the maximum load, A_c is the contact area (nm^2), h_c is the contact depth (nm) and H is the hardness of the nanocomposite film.

$$\frac{1}{E_r} = \frac{1-v^2}{E} + \frac{1-v_i^2}{E_i} \quad (3)$$

where E_r is the reduced modulus (MPa), v is the Poisson's ratio of the nanocomposite film, which was obtained by assuming that the material is isotropic in nature with the elastic modulus

evenly distributed in all crystallographic directions = 0.5, ν_i is the Poisson's ratio of the diamond indenter = 0.25, E_i is the elastic modulus of the diamond indenter = 1140 GPa and E is the elastic modulus of the nanocomposite film [6, 7].

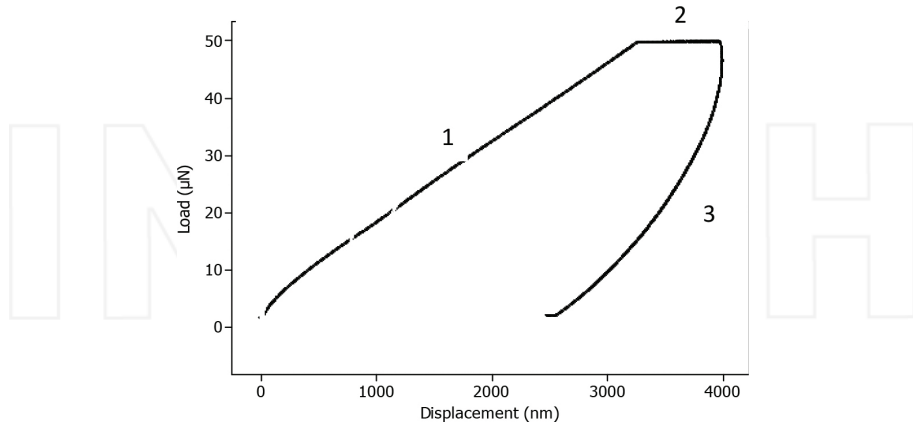


Figure 1. Load-displacement profile of a typical nanocomposite film (1, loading stage; 2, holding stage; 3, unloading stage).

3. Results and discussion

3.1. Response of cassava starch-zinc nanocomposite film to dynamic loading

The responses of the thickness and the various concentrations of glycerol and zinc nanoparticles on the rheological behaviour of the cassava starch-zinc nanocomposite films to dynamic loading are shown in **Figures 2–10**. It can be seen that the nanocomposite films behave differently under the various glycerol concentration and zinc nanoparticles at 15, 16 and 17 μm . The holding rate sensitivity of the nanocomposite film, which corresponds to the creep behaviour, was more pronounced in the 16 and 17 μm of the 45% glycerol film than the 15 μm counterpart, as shown in **Figure 2**. This may suggest that the strain rate sensitivity of the 15 μm nanocomposite film, which is a quantitative representation of a film's ability to stretch and the amount of energy absorbed when stretched [12], was lower than the other two nanocomposite films with 45% glycerol and 0% zinc nanoparticles formulation. The addition of 1% zinc nanoparticles in the formulation abruptly increased the strain rate sensitivity of the 15 μm film, as can be seen in **Figure 3**. The presence of the nanoparticles in the 15 μm film formulation might have caused an improvement in the ability of the material to withstand dead load, as exhibited by its high holding rate sensitivity in comparison to the other two thicknesses. The responses of the nanocomposite films to dynamic loading were somewhat conspicuous with a further increase in the concentration of the zinc nanoparticles to the matrix of the 45% glycerol films (**Figure 4**). It can be seen that the 15 μm film responded to dynamic

loading and strain rate sensitivity than both the 16 and 17 μm films due probably to its lower brittle nature. Sanyang et al. [13] observed similar decrease in the strain energy of biodegradable films based on sugar palm at 45% glycerol concentration. It is possible that the high amount of the dry matter in the matrix of the films, which may be associated with the increased thickness [9], might have been responsible for the higher brittle nature and hence the poor responses to dynamic loading of the 16 and 17 μm films. Interestingly, the responses of the nanocomposite films containing 50% glycerol at the various concentrations of the zinc nanoparticles and thicknesses were slightly different from those containing 45% glycerol with respect to the strain rate sensitivity (Figures 5–7). For instance, the nanocomposite films responded to dynamic loading almost in the same manner irrespective of their distinct thickness, as shown in Figure 5. It is likely that the 10% increment in the glycerol concentration might have led to the improvement in the strain rate sensitivities of the nanocomposite films.

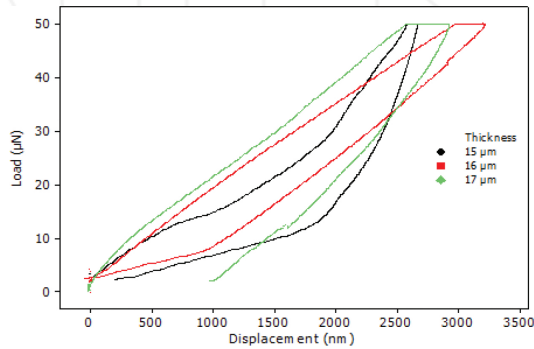


Figure 2. Response of the 45% glycerol–0% zinc nanocomposite films of different thickness to dynamic loading causing wear.

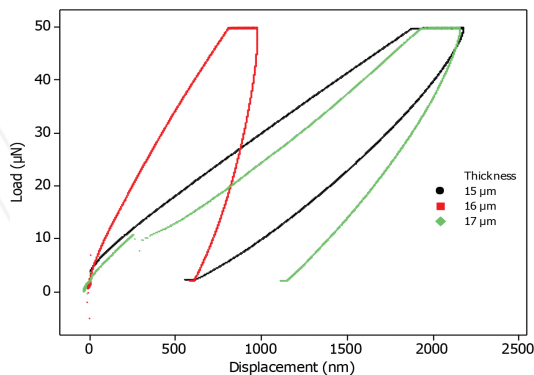


Figure 3. Response of the 45% glycerol–1% zinc nanocomposite films of different thickness to dynamic loading causing wear.

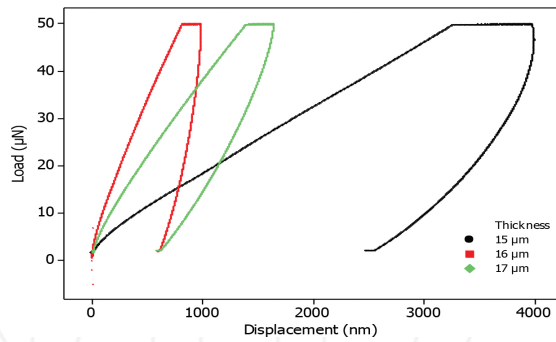


Figure 4. Response of the 45% glycerol-2% zinc nanocomposite films of different thickness to dynamic loading causing wear.

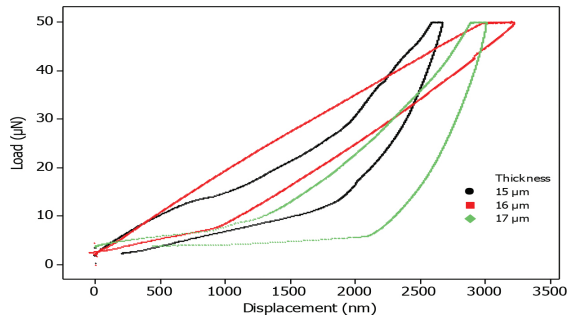


Figure 5. Response of the 50% glycerol-0% zinc nanocomposite films of different thickness to dynamic loading causing wear.

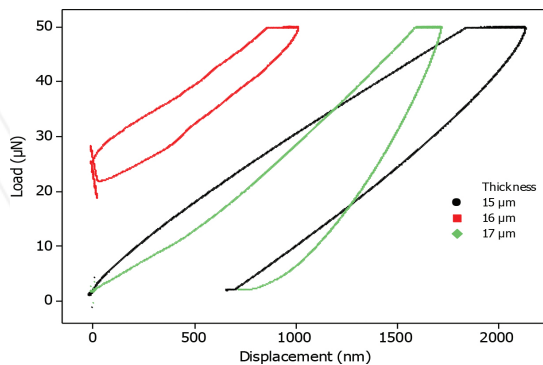


Figure 6. Response of the 50% glycerol-1% zinc nanocomposite films of different thickness to dynamic loading causing wear.

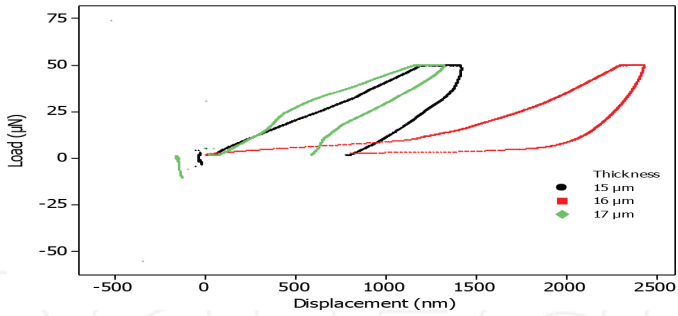


Figure 7. Response of the 50% glycerol-2% zinc nanocomposite films of different thickness to dynamic loading causing wear.

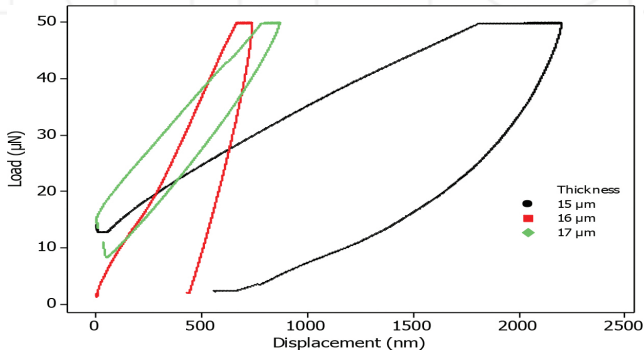


Figure 8. Response of the 55% glycerol-0% zinc nanocomposite films of different thickness to dynamic loading causing wear.

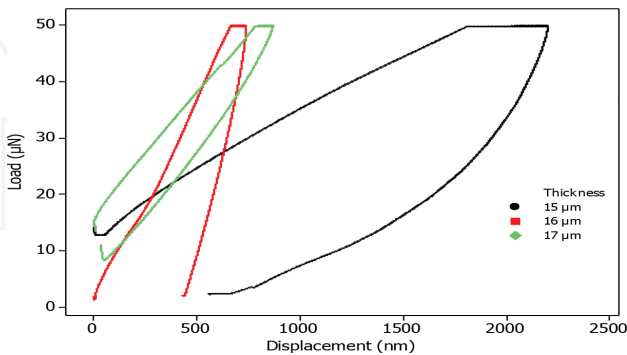


Figure 9. Response of the 55% glycerol-1% zinc nanocomposite films of different thickness to dynamic loading causing wear.

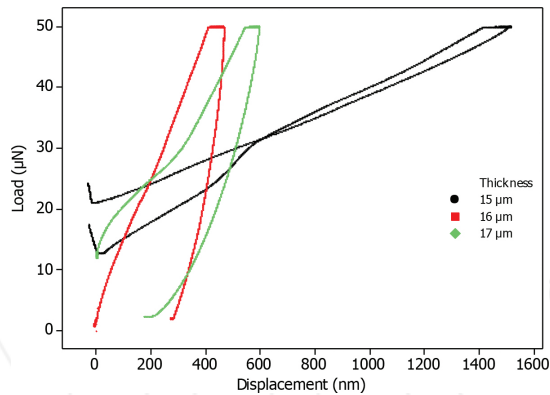


Figure 10. Response of the 55% glycerol-2% zinc nanocomposite films of different thickness to dynamic loading causing wear.

The addition of 1% zinc nanoparticles might have caused the nanocomposite films to behave differently (Figure 6), with the 16 µm film responding inconsistently to dynamic loading with a further increase in the concentration of the nanoparticles (Figure 7). The addition of 1% zinc nanoparticles to the nanocomposite films containing 55% glycerol does not show any improvement from those with 0% of the nanoparticles, as shown in Figures 8 and 9. An improvement was noticed when 2% zinc nanoparticle was used in the film formulation; where the strain rate sensitivity was considerably lower (Figure 10). As pointed out previously, here again the 15 µm film responded considerably to dynamic loading than the other two counterpart films. Tall et al. [11], in a similar research noted the response of Ni-Ti thin films to dynamic loading and unloading with distinct strain rate sensitivity. Additionally, the strain energy released during the loading part of the hysteresis loop is in the range of 40–190 pNm, and this varies with thickness and glycerol concentration (Figures 2–10). In all cases, the energy dissipated was recovered into the system during the unloading part of the loop. Also, the energy dissipated by the nanocomposite films was lower than those with 0% zinc nanoparticles, irrespective of the concentration of the glycerol but decreases with the thickness of the films. This corroborates the works of Tall et al. [11] who observed regions of loading and unloading of the hysteresis loop of the Ni-Ti thin films. Also, regions of loading and unloading were observed in Cu₂O thin films [7]. This behaviour might be associated with the increased displacement of the film with a slight change in the applied load as the concentration of the glycerol and zinc nanoparticles increased. The rheological properties are important parameters for the formation, application and quality of the films. The measure of wear and tear of the films, and its knowledge informs the engineer on the choice of appropriate films to withstand high mechanical load at nanoscale, especially during transportation on rough roads.

3.2. Effects of the processing parameters on rheological properties of the films

The effects of the processing parameters (thickness, zinc nanoparticles and glycerol concentration) on the rheological properties of the cassava starch nanocomposite films are shown in

Figures 11 and 12. It can be seen that the hardness and Young's modulus varied inconsistently with the concentrations of glycerol and zinc nanoparticles at 15, 16 and 17 μm thickness (**Figure 11**). This implies that an increase in the applied load does not necessarily reduce the area of cross-section due to the influence exerted by the zinc nanoparticles and the glycerol on the crystalline lattice of the films. The elastic and plastic works, which determines the plasticity indices of the nanocomposite films, decreased generally with the thickness, zinc nanoparticles and glycerol concentration (**Figure 12**). Higher plasticity index was obtained for 15 μm film than 16 and 17 μm films irrespective of the concentrations of glycerol and zinc nanoparticles. It is likely that the high amount of the dry matter in the matrix of the films, which is associated with the increased thickness [9], might have been responsible for the decreased elastic and plastic works of the 16 and 17 μm films. Jorge et al. [14], who studied the mechanical properties of gelatine nanocomposite films and investigated the effect of montmorillonite concentration on the properties, corroborated our findings. The authors revealed that the hardness and Young's modulus of the films were inconsistent with montmorillonite concentration, thus indicating the reinforcement of the film matrix by the nanoparticle. The lower values of plasticity indices of the films might also be associated with the plasticising effect of the absorbed glycerol during formulation. Thus, the zinc nanoparticles can be said to enhance the rheological properties, particularly stress and Young's modulus for high speed packaging application [15].

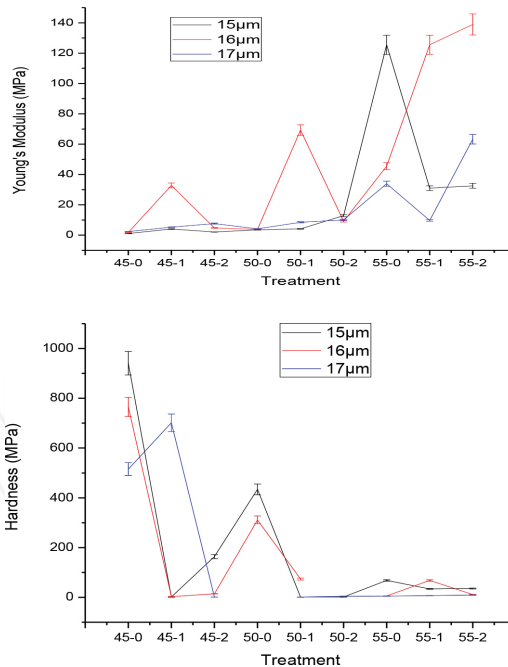


Figure 11. Effect of experimental variables on creep and Young's modulus of the films.

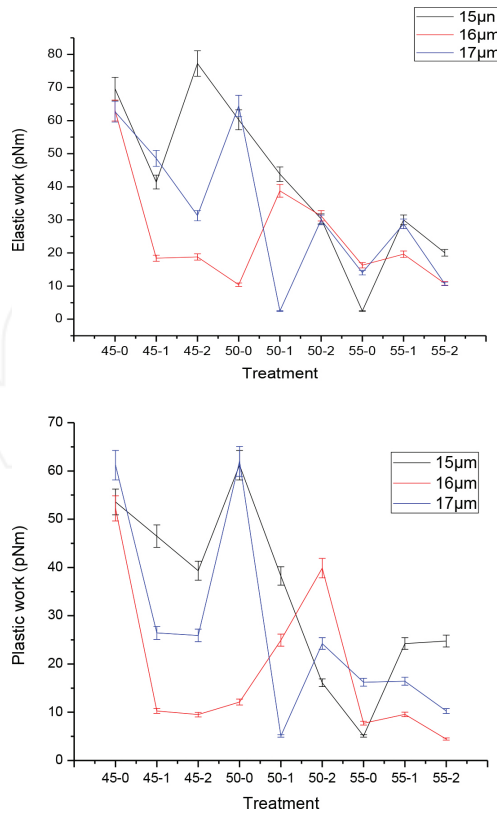


Figure 12. Effect of experimental variables on the elastic and plastic works of the films.

There is a correlation between the elastic work done during dynamic loading and the viscoelasticity of the nanocomposite films [16–18]. Thus, since the elastic work is affected by the concentration of glycerol and thickness of the film formulation, the viscoelastic behaviour of the nanocomposite film might as well be influenced. It is possible that the increased thickness of the nanocomposite films, which can affect their flexibility, might be associated with their poor viscoelasticity. Therefore, in order to adapt to the possible deformation or wear, occurring especially on rough roads [19, 20], the viscoelasticity of the nanocomposite films must be high enough so that the elastic work done during the dynamic loading is subsequently recovered in the unloading part of the cycle.

4. Conclusions

The dynamic behaviour of nanocomposite film, which is associated with viscoelastic behaviour, has not being well understood and characterised. This has often caused wears and

consequent damages of the packaging materials, particularly during the handling and transportation of food. Nanoindentation provides a convenient tool for probing the basic rheological properties and the extent of stress-induced phase transition in small volumes of nanocomposite materials. Thus, this research was carried out to determine the nano-rheological behaviour of cassava starch-zinc nanocomposite films under dynamic loading for high speed transportation of packaged food. The films, whose dried thicknesses were in the range of 15–16 μm , were prepared by casting mixtures of 24 g of cassava starch, 45–55% w/w of glycerol and 0–2% w/w of zinc nanoparticles. The responses of cassava starch-zinc nanocomposite films to dynamic loading were determined, thus assessing their suitability for high speed packaging application. The increase in the concentrations of glycerol (>45%) and zinc nanoparticles (>1%) in the formulation might be responsible for the increased strain rate sensitivity of the 15 μm film. The hardness and Young's modulus of the films varied inconsistently with the concentrations of glycerol and zinc nanoparticles at 15, 16 and 17 μm thickness, because of the increased displacement with a slight change in the applied load. The strain energy absorbed was lower for the 15 μm film, which absorbed 40 pNm during the loading part of the hysteresis loop, than for the 16 and 17 μm films. Also, only 0.5 pNm of the strain energy was finally dissipated during the unloading part of the loop. The creep response of the 15 μm film makes it viscoelastic enough to withstand death loads or wear at higher loads. Consequently, the film can be used for the high speed packaging of food and food products.

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Author details

Adeshina Fadeyibi^{1*}, Zinash D. Osunde², Gbabo Agidi², Evans C. Egwim³ and Peter A. Idah²

*Address all correspondence to: adeshina.fadeyibi@kwasu.edu.ng

1 Department of Agricultural and Biological Engineering, Kwara State University Malete, Nigeria

2 Department of Agricultural and Bioresources Engineering, Federal University of Technology Minna, Minna, Nigeria

3 Department of Biochemistry, Federal University of Technology Minna, Minna, Nigeria

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