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Prediction of Some Physical Attributes of Cassava Starch–Zinc Nanocomposite Film for Food-Packaging Applications

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Abstract

Characterization of nanocomposite film requires a high level of instrumentation and accuracy in measurements. It is normally arduous to achieve correct measurement of a system under different conditions using the same instrument without allowing for errors. For this reason, model representation of a system is usually encouraged. In this research, empirical model equations were developed for predicting some physical attributes of cassava starch–zinc nanocomposite film for food-packaging applications. Samples of the films, with thickness ranging between 15 and 17 μm , were developed by blending 24 g of cassava starch, 0–2% zinc nanoparticles, and 45–55% glycerol. The permeability of the films, which helps in maintaining the quality of packaged food, was determined due to oxygen and water vapour at a temperature of 27 °C and 65% RH. Elastic modulus and hardness were determined using nano-indentation techniques. Empirical model equations were developed using Box–Behnken design from 60% of the total data and the remainder were predicted. Results showed that the models developed are fit, and there were no significant differences between the 40% remaining data and model predicted data (<0.05). The contributions of the model terms to the validity of the equations were generally high with mean square error (MSE) $<10\%$. The result indicates that the models can be suitable for predicting permeability, hardness, and elastic modulus of cassava starch–zinc nanocomposite film.

Keywords Elastic modulus · Hardness · Modelling · Cassava starch · Zinc nanocomposite · Packaging

Introduction

Application of biofilms in the packaging of food and food products has led to the renewed impetus of replacing non-degradable synthetic polymers with biodegradable materials recently. In many industrial food applications, biodegradable films have been regarded with high potential, especially in prolonging the shelf life of food. Success attained in the use of the films as packaging materials for food may be partly due to the ease with which the films are decomposed or degraded by the actions of living organisms such bacteria, yeast, and fungi, which make them environmentally friendly. In addition, partly responsible are the high water vapour and low gas permeability of this class of packaging materials,

which is an advantage, especially when use in prolonging the shelf life of food products. Biodegradable materials are comparable to the synthetic polymers in terms of strength, elastic work, and creep behaviours [6]. Permeability and mechanical behaviour of the nanocomposite film are important in deciding its overall suitability for food-packaging application. Thus, knowledge of these properties is important in understanding the behaviour of the film, especially during transportation of packaged foods on rough roads.

Physical properties of the nanocomposite films, including permeability due to oxygen and water vapour and mechanical behaviour, have been reported [1, 7, 12]. For instance, Ali and Noori [1] and Suppakul et al. [12] reported a decrease in oxygen and water vapour permeabilities of biodegradable films with an increase in nanoparticles and plasticizer concentrations. Mechanical properties such as elastic modulus and hardness are important in understanding the behaviour of nanocomposite materials under dynamic loading, especially in high-speed transportation [7]. In addition, the degree of degradation of the materials can be measured with a fair knowledge of the mechanical properties [4, 10]

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and this can help in selecting appropriate materials for a specific packaging application [11]. Elastic modulus of biodegradable film is an essential parameter for understanding the stiffness of the material at ambient atmospheric condition. This in turn plays a role in its application as packaging material for fruits and vegetables [3]. For instance, stiffer biodegradable films may not be suitable for rigid agricultural products, like okra, but may be ideal material for packaging less rigid products. As much as possible, the desired stiffness of the film must be determined to meet the packaging requirement of the product. Despite the benefits of using the biodegradable nanocomposite film, especially in food packaging, the rigour involved in its processing, scarcity of measuring instrument, and the high cost of instrumentation in measurement of properties have rendered it somewhat unattractive.

It is possible to predict the physical behaviour of the nanocomposite film without necessarily subjecting them to testing. Amounts of nanoparticles, plasticizer, and thickness in the matrix of the film, as process variables, may ultimately decide its physical behaviour when used as food-packaging material. It is, therefore, necessary to establish an empirical relationship which describes the physical behaviour of biodegradable film with respect to the process variables. At the moment, there is no reported research on modelling of the physical behaviour of the nanocomposite food-packaging materials. The objective of this research, therefore, was to develop empirical model suitable for predicting the physical behaviour of the cassava starch–zinc nanocomposite film, including oxygen permeability, water vapour permeability, hardness, and elastic modulus for food-packaging application.

Method

Development of Cassava Starch–Zinc Nanocomposite Film

Nanocomposite films were developed using the method described by Fadeyibi et al. [5]. In this approach, mixtures of 24 g of cassava starch (2%, wb), zinc nanoparticles (4–6 nm particle size in the range of 0–2%), and glycerol of analytical grade (45–55%) in 600 ml distilled [7]. Resulting solutions were poured in plastic moulds of 8, 10, and 12 mm depths for casting and thereafter dried to 4% moisture content (db) at 95 °C and 60% RH for 24 h. Dried thicknesses of the film were measured using a plastic micrometer. The mould of 8 mm depth gave an average dried thickness of $15.14 \pm 0.22 \mu\text{m}$, while those with 10 mm and 12 mm depths were $16.21 \pm 0.36 \mu\text{m}$ and $17.38 \pm 0.13 \mu\text{m}$, respectively. Thus, 27 samples were prepared from a combination of zinc

nanoparticles, glycerol concentration, and thickness of the nanocomposite film at three levels each.

Characterization of Cassava Starch–Zinc Nanocomposite Film

ASTM standard [2] was used to determine water vapour permeability at a temperature and a relative humidity of 27% and 65%, respectively. Column absorption method described by Fadeyibi et al. [6] was used to determine the oxygen permeability of the cassava starch–zinc nanocomposite film. Nano-indentation technique was used to determine the mechanical response based on hardness and elastic modulus of the films [13]. A load–displacement response of each sample (Fig. 1), which described the area of loading or energy dissipation and unloading or energy recovery, was obtained from the instrument, and thereafter, values of hardness and elastic modulus of the films were computed.

Model Development and Analysis

A quadratic polynomial regression model was assumed for predicting (Y) response variable. Other forms of the polynomial regression models were tested against the chosen quadratic model before it was accepted for use. Model of the form in Eq. (1) was developed to describe the response (Y):

$$Y = \beta_0 + \sum_{n=1}^{26} \beta_n(X_1, X_2, X_3), \quad (1)$$

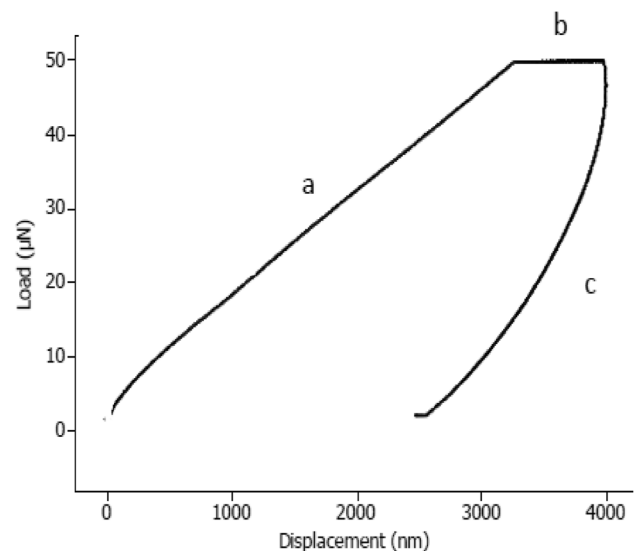


Fig. 1 Typical load–displacement profile of cassava starch–zinc nanocomposite film (a energy dissipation, b energy recovery, c creep) [7]

where Y is the value of the considered experimental predicted response variable (physical attributes of the films), X_1 , X_2 , and X_3 are the values of thickness of the film, concentrations of glycerol, and zinc nanoparticles, respectively, β_0 is the intercept value, and β_n takes the value of interaction, quadratic, and linear coefficients.

Box–Behnken Design (BBD) was employed to generate the models from 60% of the total data from 27 samples of the experiment. Thus, 17 randomly selected data from each response were used in BBD, while the remaining ten data from the responses were kept for model validation. Layout of the combinations of the process variables and the physical attributes of the nanocomposite films in the BBD protocol, with individual experiments carried out in random order, is shown in Table 1.

Fitting of Data to the Model and Validation

Experimental data were fitted to the regression models generated using Design Expert Package (ver. 10.0). The degree of fitness of the models was determined by both R^2 and R^2_{adj} . The resulting models were used to predict the remaining 40% of the data and the results were compared with the experimented at $p < 0.05$ using the mean square error approach in the following equation:

$$MSE = \frac{1}{n} \sum_{n=1}^n (Y_e - Y_p)^2, \quad (2)$$

where MSE = mean square error (square of unit of Y), Y_e = experimented response, Y_p = model predicted response, and n = total number of samples (27).

Analysis of Variance of Model

Analysis of variance (ANOVA) of the developed models was carried out to investigate the significance of the model terms at $p \leq 0.05$. In the event that the model terms are not significant, the model was improved by stepwise regression or backward elimination regression procedures, as the case may be to remove the insignificant terms of the models [9, 14]. This was also carried out using the Design Expert Software Package (ver. 10.0).

Results and Discussion

Prediction and Validation of Permeability of the Films

Model equations for predicting the oxygen and water vapour permeabilities of the cassava starch–zinc nanocomposite at a temperature of 27 °C and 65% relative humidity were fitted in Eqs. (3) and (4). The analyses of variance of the models showing the effects of the variables and their interactions are shown in Table 2:

Table 1 Combination of process variables in Box–Behnken design

Sdt	Run	Process variables				Physical attributes		
		T_f (μm)	C_g (%)	C_{np} (%)	H (kPa)	E (MPa)	OP (g/m Pa.s)	WVP (g/m Pa.s)
15	1	16	55	1	++	++	++	++
3	2	15	55	1	++	++	++	++
5	3	15	50	0	++	++	++	++
7	4	15	50	2	++	++	++	++
13	5	16	50	1	++	++	++	++
2	6	17	45	1	++	++	++	++
11	7	16	45	2	++	++	++	++
1	8	15	45	1	++	++	++	++
17	9	16	50	1	++	++	++	++
9	10	16	45	0	++	++	++	++
14	11	16	50	1	++	++	++	++
16	12	16	50	1	++	++	++	++
4	13	17	55	1	++	++	++	++
6	14	17	50	0	++	++	++	++
10	15	16	55	0	++	++	++	++
12	16	16	55	2	++	++	++	++
8	17	17	50	2	++	++	++	++

t_f film thickness, C_g concentration of glycerol, C_{np} concentration zinc nanoparticles, H hardness, E elastic modulus, OP oxygen permeability, WVP water vapour permeability

Table 2 Analysis of variance for models of the permeability of the films

Parameter	Source	Model effect	Sum of squares	df	Mean square	F value	p value (prob > F)
WVP	Model	4.97×10^{-9}	8.95×10^{-20}	9	9.94×10^{-21}	27.55072*	0.0001
	A-film thickness	-8.3×10^{-10}	7.61×10^{-20}	1	7.61×10^{-20}	210.954	<0.0001
	B-glycerol conc	9.7×10^{-11}	7.45×10^{-21}	1	7.45×10^{-21}	20.65592	0.0027
	C-conc of Np	5.3×10^{-11}	3.25×10^{-22}	1	3.25×10^{-22}	0.900935	0.3741
	AB	-3.2×10^{-12}	1.05×10^{-21}	1	1.05×10^{-21}	2.917915	0.1314
	AC	$-3.30E-12$	4.29×10^{-23}	1	4.29×10^{-23}	0.118885	0.7404
	BC	-4.4×10^{-13}	1.89×10^{-23}	1	1.89×10^{-23}	0.052435	0.8254
	A ²	2.8×10^{-11}	3.28×10^{-21}	1	3.28×10^{-21}	9.090276	0.0195
	B ²	-3.8×10^{-13}	3.87×10^{-22}	1	3.87×10^{-22}	1.072483	0.3348
	C ²	1.4×10^{-11}	7.74×10^{-22}	1	7.74×10^{-22}	2.146145	0.1863
	Residual		2.53×10^{-21}	7	3.61×10^{-22}		
	Lack of fit		2.53×10^{-21}	3	8.42×10^{-22}		
	Pure error		0	4	0		
	Cor total		9.20×10^{-20}	16			
OP	Model	3.7×10^{-10}	3.7×10^{-23}	1	3.67×10^{-23}	90.95665*	<0.0001
	A-film thickness	-3.8×10^{-11}	1.1×10^{-22}	1	1.06×10^{-22}	261.4593	<0.0001
	B-glycerol conc	-9.4×10^{-13}	1.3×10^{-23}	1	1.29×10^{-23}	31.83382	0.0008
	C-conc of Np	1.4×10^{-11}	2.5×10^{-27}	1	2.50×10^{-27}	0.006192	0.9395
	AB	-5.0×10^{-15}	5.5×10^{-24}	1	5.52×10^{-24}	13.6785	0.0077
	AC	-1.2×10^{-12}	8.4×10^{-26}	1	8.41×10^{-26}	0.208305	0.6619
	BC	2.9×10^{-14}	5.9×10^{-24}	1	5.86×10^{-24}	14.52122	0.0066
	A ²	1.2×10^{-12}	1.8×10^{-26}	1	1.78×10^{-26}	0.044062	0.8397
	B ²	2.6×10^{-15}	3.7×10^{-24}	1	3.72×10^{-24}	9.214991	0.019
	C ²	9.4×10^{-13}	2.8×10^{-24}	7	4.04×10^{-25}		
	Residual		2.8×10^{-24}	3	9.42×10^{-25}		
	Lack of fit		0	4	0		

df degree of freedom, WVP water vapour permeability, OP oxygen permeability, NP nanoparticles

*Significant when p value [$\text{prob} > F$] < 5%

$$\text{a) } W_{vp} = \left[49.7 - 8.25t_f + 0.99c_g + 0.53c_{np} - 0.032t_f \cdot c_g - 0.033t_f \cdot c_{np} - 0.004c_g \cdot c_{np} + 0.279t_f^2 - 0.004c_g^2 + 0.136c_{np}^2 \right] [\times 10^{-10}] \quad \begin{cases} R^2 = 0.9448 \\ R_{Adj}^2 = 0.9320 \\ \text{MSE} = 0.005 \end{cases} \quad (3)$$

$$\text{b) } W_{vp} = \left[3689.02 - 384.8t_f - 9.36c_g + 142.03c_{np} - 0.05t_f \cdot c_g - 11.8t_f \cdot c_{np} + 0.29c_g \cdot c_{np} + 11.8t_f^2 + 0.003c_g^2 + 9.4c_{np}^2 \right] [\times 10^{-13}] \quad \begin{cases} R^2 = 0.9837 \\ R_{Adj}^2 = 0.9628 \\ \text{MSE} = 0.004 \end{cases} \quad (4)$$

where c_{np} = concentration of nanoparticles (%), c_g = concentration of glycerol(%), t_f = thickness of the film (μm), WPR = water vapour transmission rate (g/m Pa.s), and OP = oxygen permeability (g/m Pa.s).

Equations (3) and (4) are quadratic models for predicting oxygen and water vapour permeabilities of the cas-sava starch–zinc nanocomposite film. The contributions

of glycerol concentration and thickness on the overall acceptability of Eq. (3) were high, while the interaction and quadratic terms of the model only offered negative effects. An increase in thickness and glycerol concentration may, therefore, increase the water vapour permeability of the resulting nanocomposite film. The contribution of the quadratic and interaction terms to the validity of Eq. (4) was

also high, while the glycerol concentration and the thickness offered individual negative effects. This may mean that an increase in the thickness and the glycerol concentration will lead to a decrease in the oxygen permeability of the film. Corroborating this investigation, Fadeyibi et al. [6] reported that the thickness and the concentration of glycerol significantly affect the performance of the biodegradable film in their work on development and optimization of cassava starch–zinc nanocomposite films for potential application in food packaging. It can be seen that the values of the actual and adjusted coefficients of determination were high and not too far apart from each other. This may mean that the experimented data used in generating the models are fit and can adequately predict the values of oxygen and water vapour permeability. Analysis of variance of the model terms showed that all the individual effects of the variable terms and quadratic effect of the thickness have

a significant contribution to the performance of Eq. (3) at $p < 0.05$ (Table 2). It can also be seen from the analysis of variance that Eq. (4) was adequate around all the variables, including their interaction and quadratic terms. Furthermore, the results presented in Fig. 2 showed that the models developed are valid as they are able to predict the values closer to the experimented with an approximate MSE value of 0.005.

Prediction and Validations of the Mechanical Properties of the Films

Relationships between the responded mechanical properties obtained from the nano-indentation test and the experimental variables are expressed in Eqs. (5) and (6). The analyses of variance of the models, occurring as a result of differences between treated variables and their interactions, are shown in Table 3:

$$a) \log_{10} M_e = -214.8 + 21.6t_f + 1.64c_g + 1.55c_{np} - 0.031t_f \cdot c_g - 0.045t_f \cdot c_{np} - 0.627t_f^2 - 0.01c_g^2 - 0.39c_{np}^2 \quad \begin{cases} R^2 = 0.9343 \\ R_{Adj}^2 = 0.8498 \\ \text{MSE} = 0.049 \end{cases} \quad (5)$$

$$b) H^{1/2} = -1049.70 + 74.019t_f + 20.55c_g - 69.50c_{np} - 1.43t_f \cdot c_g + 1.25c_g \cdot c_{np} \quad \begin{cases} R^2 = 0.9026 \\ R_{Adj}^2 = 0.8585 \\ \text{MSE} = 0.028 \end{cases} \quad (6)$$

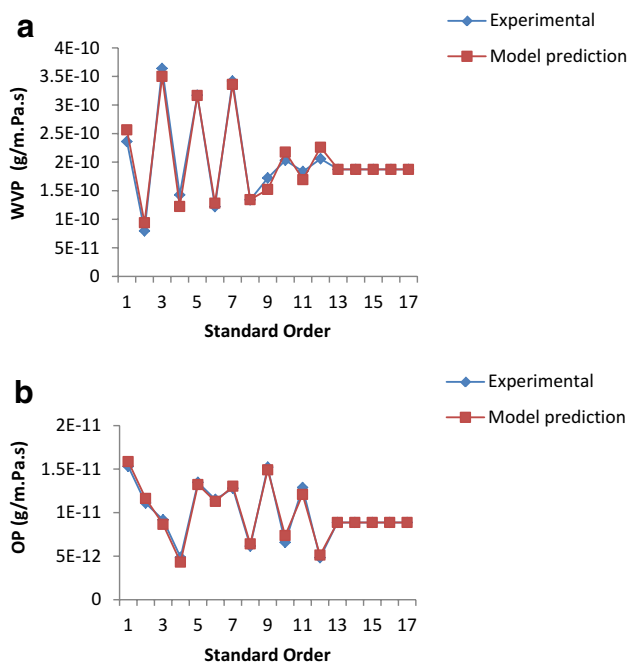


Fig. 2 Validation of the predictive model of Cassava starch nanocomposite films: **a** water vapour permeability and **b** oxygen permeability

where c_{np} = concentration of zinc nanoparticles, c_g = concentration of glycerol, t_f = thickness of the film (μm), M_e = elastic modulus (MPa), and H = hardness (kPa).

A quadratic expression, with the logarithm to base 10 transformation, was developed for predicting the elastic modulus with higher actual and adjusted coefficients of determination ($R^2 = 93.4\%$, $R_{adj}^2 = 84.98\%$), as shown in Eq. (5). It can be seen that the thickness, glycerol, and zinc nanoparticles have greater influence on the validity of Eq. (5) than their respective quadratic and interaction model terms. An increase in the thickness, concentrations of glycerol, and zinc nanoparticles will ultimately lead to an increase in the elastic modulus. Lagaron and Sanchez-Garcia [8], in a similar research on thermoplastic nanobiocomposites for rigid and flexible food-packaging applications, reported an increase in the elastic modulus of nanocomposite film with an increase in the concentration of nanoparticles and the plasticizer. A second function interaction equation (2FI), with a square root transformation, was developed for predicting the hardness of the film. A higher actual and adjusted coefficients of determination ($R^2 = 90.3\%$, $R_{adj}^2 = 85.9\%$) were observed in this case, as shown in Eq. (6). The contributions of the thickness and the glycerol concentration on the

Table 3 Analysis of variance of the model for elastic modulus and hardness of nanocomposite film

Parameter	Source	Model effects	Sum of squares	<i>df</i>	Mean square	<i>F</i> value	<i>p</i> value (prob > <i>F</i>)
Elastic modulus	Model	− 214.77	5.400	9	0.600	11.06*	0.0023
	A-film thickness	21.5937	0.022	1	0.022	0.410	0.5429
	B-glycerol conc	1.64055	2.010	1	2.010	37.00	0.0005
	C-conc of Np	1.54711	0.420	1	0.420	7.740	0.0272
	AB	− 0.0309	0.096	1	0.096	1.760	0.2259
	AC	− 0.0452	8.2×10^{-3}	1	8.2×10^{-3}	0.150	0.7096
	BC	3.8×10^{-3}	1.4×10^{-3}	1	1.4×10^{-3}	0.026	0.8762
	A ²	− 0.6267	1.650	1	1.650	30.49	0.0009
	B ²	− 0.0104	0.290	1	0.290	5.340	0.0541
	C ²	− 0.3918	0.650	1	0.650	11.91	0.0107
	Residual		0.380	7	0.054		
	Lack of fit		0.380	3	0.130		
	Pure error		0.000	4	0.000		
	Cor total		5.780	16			
Hardness	Model	− 1049.701	1069.11	5	213.82	20.41*	< 0.0001
	A-film thickness	74.018769	46.28	1	46.28	4.42	0.0594
	B-glycerol conc	20.552695	249.69	1	249.69	23.84	0.0005
	C-conc of Np	− 69.49917	412.66	1	412.66	39.39	< 0.0001
	AB	− 1.432274	205.14	1	205.14	19.58	0.0010
	BC	1.2463412	155.34	1	155.34	14.83	0.0027
	Residual	− 1049.701	115.23	11	10.48		
	Lack of fit		115.23	7	16.46		
	Pure error		0.000	4	0.000		
	Cor total		1184.34	16			

df degree of freedom, *NP* nanoparticles

*Significant when *p* value [prob > *F*] < 5%

overall acceptability of Eq. (6) were greater than the combined effects of the zinc nanoparticles, quadratic and interaction terms of the model. This implies that the high hardness of the nanocomposite film was a result of the greater influence of the thickness and the glycerol concentration. According to Fadeyibi et al. [6], thickness and glycerol content of the nanocomposites film in excess of 17 μm and 55%, respectively, may cause the material to be brittle and obsolesce for food packaging. As much as possible, therefore, the requirement is to make the film less hard and suitable for the intended application. So long as the thickness and concentration of glycerol does not exceed the stated limits, the model equation can predict the hardness reliably well. In addition, the influences of the individual effects, interaction, and quadratic terms in all the models generated for elastic modulus and hardness were adequate and significant at $p < 0.05$. There were no significant differences between the experimented and model predicted data for the elastic modulus and hardness of the nanocomposite films, as shown in Fig. 3.

Conclusions

Empirical model equations were developed for predicting oxygen permeability, water vapour permeability, elastic modulus, and hardness of cassava starch–zinc nanocomposite film. About 60% of the total data used in developing the models are fit, and there were no significant differences between the experimented and model predicted data ($p < 0.05$). In addition, the models were quite general as they were able to predict the remaining 40% of the response data with mean square error (MSE) < 10%. The models may, therefore, be suitable for predicting the oxygen permeability, water vapour permeability, hardness, and elastic modulus of cassava starch–zinc nanocomposite film.

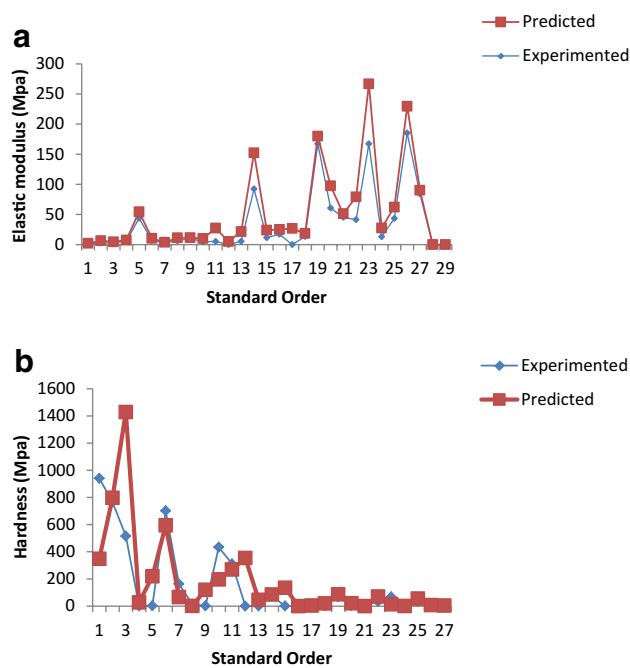


Fig. 3 Validation of the predictive model of Cassava starch nanocomposite films: **a** elastic modulus and **b** hardness

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