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Morphological and Tensile Behaviors of Carboxymethyl Cellulose-Pectin Films Reinforced with Cassava Lignin Fibre for Food Packaging

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ABSTRACT

The increasing demand for sustainable materials has driven research to renewable, easily accessible and utilization of agricultural waste. Carboxyl methylcellulose-pectin nanocomposites reinforced with cassava lignin through solution casting technique were fabricated for use in food packaging. The effects of the concentration of cassava lignin, pectin, and carboxyl methylcellulose on the tensile strength and elongation of produced films were investigated and optimized using central composite design. The morphological property was analyzed using scanning electron microscope. The result showed that addition of cassava lignin improved the tensile strength of the carboxyl methylcellulose-pectin film by 45.51% when compared with unreinforced carboxyl methylcellulose-pectin film. This indicates homogenous dispersion and distribution of the particles, as well as favorable interfacial interaction between the particles and matrix as shown in the microstructural images. Lignin fibre decreased the elongation at break of the produced films. The 1.2 wt. % of cassava lignin reinforcements show that elongation at break (EB) decreased from 70.3% to 55.4%. This could be as a result of the stiffness introduced by cassava lignin nanofiller through the creation of three-dimensional network structure in the nanocomposite film.

1. Introduction

Conventionally, food packaging products are produced from synthetic polymers derived from petrochemicals and additives derived from non-degradable, non-renewable, expensive petroleum resources. Currently, this is raising a lot of concerns with respect to both health and environmental pollution due to the non-degradability of these polymers and the emission of toxic gases during their manufacture [1-5]. In addition, the depletion of petrochemical resources and the escalating cost of synthetic polymer products constitute a significant drawback to the sustainability of the food packaging industry, particularly in Nigeria [4-8]. Recently, attention has turned to bio-derived polymers as researchers seek to develop alternative raw materials that are inexpensive, sustainable, renewable bio-degradable, low toxicity and carbon emission which can compete with petroleum-based food packages in terms of cost and properties [3-4, 23]. Pectin, a naturally occurring polysaccharide has in recent years gained increasing importance from scientists and consumers due to its biodegradability. It is commercially extracted from citrus peels and apple pomace under mildly acidic conditions and comes in two major groups based on their degree of esterification [9-15]. Carboxymethyl cellulose (CMC) has been identified as one of the most promising cellulose derivatives due to its distinctive characteristics. Surface properties, mechanical strength, tunable hydrophilicity, viscous properties, availability and abundance of raw materials, low-cost synthesis process, and other contrasting aspects make it a focus for advanced application in food, paper, textile, pharmaceutical industries, biomedical engineering, waste water treatment and storage energy production [7-10, 19-26].

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Pectins and lignin are both renewable, biodegradable, and easily accessible which make them attractive options for a range of applications [19, 24-27]. There is an obvious need for inexpensive, biodegradable, and environmentally acceptable substitutes given the high cost of synthetic polymer materials used for food packaging in Nigeria and growing worries of their health hazards and non-biodegradability [23, 26-28]. This study seeks to completely replace petroleum-based polymers used in food packaging applications with renewable and sustainable pectin, carboxymethyl cellulose and cassava lignin. The concept of blending these three bio-derived polymers is to synergistically enhance the dimensional stability and mechanical strength properties of the bio-based films for better service performance. On the other hand, the addition of bio-fillers from clay and snail shells with proven anti-flame properties would impart remarkable strength, water vapor permeability, and excellent water absorption to bio-based films.

2. Materials and Methods

2.1. Materials

Pectin, sulfuric acid, hydrochloric acid, sodium hydroxide, glycerol, ethanol, hydrogen peroxide, carboxymethyl cellulose (CMC), sodium chlorite and distilled water were sourced from the Onitsha Bridge-Head Chemical market, Anambra state. The lignin-containing cassava peel (LCCP) biomass was collected from a local cassava farm within Nnamdi Azikiwe University, Awka. The equipment and apparatuses used were stirring rod, beakers, conical flask, water bath (Model: dk -420), oven, digital weighing balance (Model: BL20001), petri-dish.

2.2. Cassava lignin extraction

Lignin-containing cassava peels (LCCP) were thoroughly washed, sun-dried for several days (Fig. 1a), ground to a fine texture and stored in plastic container at room temperature. Lignin-containing cassava peel (LCCP) was converted to nano size using 60% w/v sulfuric acid, prepared by diluting 600ml of concentrated sulfuric acid (H_2SO_4) with 400 ml of distilled water as described by Lusiana et al. (2019). The biomass (20g) was then hydrolyzed in 200ml of diluted sulfuric acid (1:10 ratio) at 60°C for 45 minutes with continuous stirring in a water bath (Fig. 1c). The mixture was removed from the bath, filtered through a fabric sieve and thoroughly washed with distilled water to remove residual acid. The resulting nano sized biomass retained on the sieve was dried in an oven at 50°C.

2.3. Nanocomposite film preparation

The films were prepared using a solution casting method similar to that described by [1]. Pectin and carboxymethyl cellulose (CMC) were separately dissolved in 75ml of distilled water under continuous stirring in a water bath for 30 minutes (Fig. 1b & 1c). The dissolved pectin and CMC solutions were then mixed, stirred and placed back in the water bath for an additional 10 minutes. Lignin-containing cassava peel (LCCP) biomass was added to the pectin/CMC solution in varying amounts each dispersed in 10ml of water. Glycerol (4.5ml) was also incorporated into the solution. The mixture was then cast into separate Petri dishes, each containing 40ml of the solution. The films were allowed to set for 96 hours (4 days) before it was transferred to a desiccator to prevent moisture absorption as shown in Fig. 1(d).

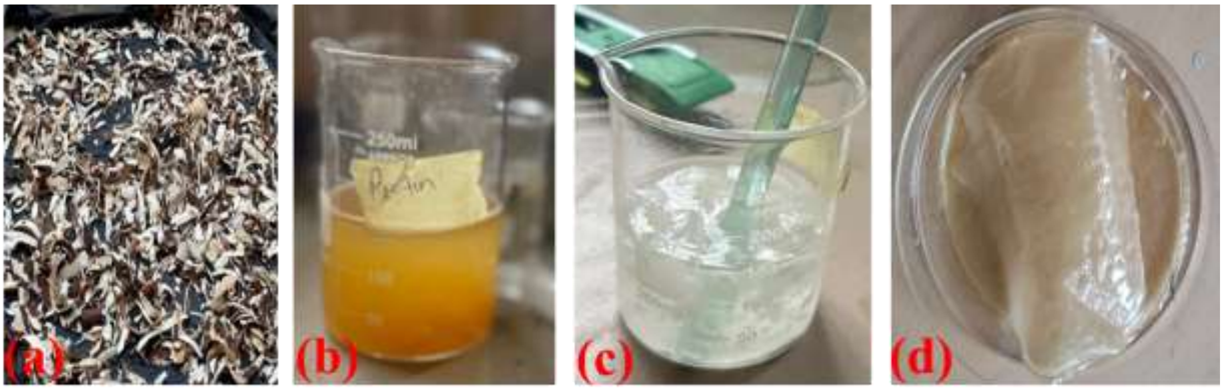

Figure 1: (a) cassava peel (b) pectin (c) mixture of pectin & cassava lignin (d) fabricated film after 96 days

2.4. Experimental design

Central composite design (CCD) of response surface methodology was used to study effects of the concentrations of lignin, pectin and carboxymethyl cellulose (CMC) as independent variables on the dependent variables (tensile strength and elongation). The factors and levels of the experimental design are displayed in Table 1. The table also showed the unit of each factor where CMC and pectin concentrations were obtained with respect to 100 g of the film forming solution, and the cassava lignin reinforcement obtained with respect to the total solid content of the film forming solution (CMC and pectin). According to earlier studies [1, 19, 22-23], the quantitative composition of the formulation was created based on the anticipated contribution of each item on the overall performance of the manufactured nanocomposite films. The CCD with an alpha (α) value of 1.68 was used to acquire the 20 experimental runs. Eight factorial points, six axial points, and six center points make up these formulas. Design Expert Software version 10 was used to examine the data, and the tests were carried out at random.

Table 1: The experimental range and levels of independent variables using Central composite design of experiment

Factor	Unit	-α	-1	0	+1	+α
CMC	g/100g	5	6.01	7.5	8.99	10
LCCP	Wt.%	0	2.03	5	7.97	10
Pectin	g/100g	5	6.01	7.5	8.99	10

2.5. Morphological analysis of nanocomposite films

The dispersion of the nano-clay/snail shell fillers within the polymer matrix was investigated using scanning electron microscopy (SEM) on a JOEL-JSM-7600F at 5kV. After being vacuum-dried for an entire night at 40 °C, the film samples were cryogenically fractured to reveal their cross section for analysis. Before examination, a 5nm coating of platinum was applied to the samples to increase contrast in the SEM image.

2.12. Mechanical analysis

The mechanical analysis of the fabricated films involving the tensile testing and elongation was carried out with M500-25CT Universal testing machine according to ASTM D882. The films were cut to 100 mm x 20 mm rectangular strips and the thickness of each sample recorded. Measurements were performed with 60 mm grip separation, pretension of 5N and crosshead speed of 50mm/min. The samples were kept in a desiccator containing calcium chloride desiccant for 24 hours prior to testing.

$$\text{Tensile Strength} = \frac{\text{Load(N)}}{\text{Thickness(mm)} \times \text{Width(mm)}} \qquad \text{(Equation. 1)}$$

$$\text{Elongation at break (\%)} = \frac{\text{Displacement at break}}{\text{Guage length}} \times 100 \qquad \text{(Equation. 2)}$$

3. Results and Discussion

3.1 Tensile strength of the nanocomposite films

Tensile strength of carboxymethyl cellulose-pectin reinforced with different amounts of cassava lignin was examined and is shown in Table 2. Enhanced tensile characteristics need favorable interfacial interactions between the polymer matrix and cassava lignin, as well as uniform dispersion and distribution of the reinforcements (Habibi et al., 2010).The tensile strength of the films was enhanced by addition of cassava lignin fibre as can be seen in Run 3 (0.14 wt% cassava lignin reinforced film), Run 5 (6.07 wt% cassava lignin reinforced film) and Run 12 (10 wt% cassava lignin reinforced film).

Table 2: Experimental design layout with actual and predicted values

Run	A: CMC g/100g	B: Pectin g/100g	C: Cassava lignin %	Tensile strength			Elongation at break		
				Actual value MPa	Predicted value MPa	Residual	Actual values %	Predicted values %	Residual
1	6.01	8.99	2.03	10.39	10.02	0.37	72.71	73.03	-0.32
2	7.50	7.50	5.00	10.96	10.74	0.22	59.8	61.56	-1.76
3	6.01	6.01	0.14	7.43	7.56	-0.21	45.8	45.56	0.24
4	8.99	8.99	1.65	12.05	11.73	0.016	68.3	67.71	0.59
5	6.01	8.99	6.07	10.01	10.05	-0.045	65.54	65.75	-0.21
6	7.50	7.50	5.00	10.76	10.74	0.020	63.78	61.56	2.22
7	7.50	7.50	5.00	10.76	10.74	0.020	62.5	61.56	0.94
8	8.99	6.01	0.75	11.76	11.89	-0.28	67.7	68.88	-1.18
9	7.50	7.50	5.00	11.06	10.74	0.32	60.51	61.56	-1.05
10	5.00	7.50	5.00	10.79	10.86	-0.066	57.4	56.70	0.70
11	7.50	10.00	5.00	8.92	9.26	-0.34	75.24	74.96	0.28
12	7.50	7.50	10.00	9.85	9.50	0.35	55.4	55.79	-0.39
13	7.50	7.50	0.00	7.23	8.39	-0.48	70.3	67.79	2.51
14	7.50	7.50	5.00	10.31	10.74	-0.43	61.2	61.56	-0.36
15	6.01	6.01	2.03	8.75	8.68	0.074	48.3	50.39	-2.09
16	10.00	7.50	1.20	14.15	13.89	-0.061	83.1	79.77	1.43
17	8.99	8.99	2.03	9.61	9.31	0.30	75.41	77.15	-1.74
18	7.50	5.00	0.25	8.65	8.26	0.21	58.75	56.90	1.85
19	7.50	7.50	5.00	10.61	10.74	-0.13	61.2	61.56	-0.36
20	8.99	6.01	2.03	10.74	10.61	0.13	74.58	75.87	-1.29

Though higher concentration of the cassava lignin showed a little decline in the tensile strength, which might have been caused by poor dispersion by further creating stress sites in the matrix and the observation aligns with the report of Agustin et al. (2013) [2]. Furthermore, the contributions of cassava lignin to the tensile strength of the nanocomposite film were observed to be more than those of carboxymethyl cellulose and pectin when the following runs; 8 and 20, 16 and 11 were closely evaluated. Similar observation was also made by Oyeka et al. (2020) [19] in their study on the preparation and characterization of polyvinyl alcohol/gelatin blends. Overall, least tensile strength observed in Run 3, followed Run 13 and Run 18 with values of 7.43MPa, 8.23MPa, and 8.65MPa respectively. Conversely, Run 16 had the highest tensile strength of 14.15 MPa on the nanocomposite film, whereas Runs 4 and 8 recorded tensile strengths of 12.05 MPa and 11.76 MPa, respectively. This result is consistent with the range reported by Mangaraj et al., (2009) [9] that low density polyethylene, which is primarily used in film packaging applications, has a tensile strength within 8–31 MPa. Additionally, Alves et al. (2015) [1] reported that the tensile strength for CNC/gelatin in corn starch plasticized films ranged from 10.91 MPa to 49.09 MPa.

3.2 Elongation at break of the nanocomposite films

Elongation at break (EB) of the cassava lignin fibre reinforced carboxymethyl cellulose/pectin nanocomposite film show a decrease in values when compared with the value of the unreinforced (Run 13) as seen in Table 2. For example, considering unreinforced film of Run 13 against Run 6 and Run 12 having 5wt% and 10wt% cassava lignin reinforcements respectively shows that EB decreased from 70.3% to 55.4%. This could have resulted from the stiffness introduced by cassava lignin nanofiller through the creation of three-dimensional network structure in the nanocomposite film [22]. Additionally, although the packaging application favors higher values of EB, it must also be proportionate to withstand strength required to carry the packing load. The contributions of each component were further evaluated through statistical analysis to obtain the optimal performance of the nanocomposite films. The maximum EB value obtained in this study was 83.1%, with the lowest value of 45.75% corresponding to Runs 16 and 3, respectively.

3.3. Statistical analysis

Multiple regression analysis of the experimental data (quadratic model) was most suitable for analyzing the independent variables in relation to the response variables of elongation at break (EB) and tensile strength (TS) as shown in Table 3.

Equations 3 (Tensile strength) and 4 (Elongation at break) represent the final model equation that was produced for predicting the responses for a given level of each factor. However, while the negative signs suggest opposing impacts on the responses, the positive coefficients demonstrate the favorable effects of the factors on the responses. It was observed that the coefficients associated with A (CMC), B (pectin), C (cassava lignin), AC (interaction between CMC and cassava lignin), BC (interaction between pectin and cassava lignin) and A^2 are positive for the tensile strength response. However, CMC and pectin only made positive contribution to the elongation at break, while the interaction between them and cassava lignin affected the EB negatively. Obviously, the highest regression coefficients are related to CMC and cassava lignin for the tensile strength while the coefficients associated with CMC and pectin are for elongation at break. Thus, it can be concluded that whereas CMC and pectin had the biggest effects on elongation at break, CMC and cassava lignin had the biggest effects on tensile strength. This is accurate since the cassava lignin can add network structure to the matrix, strengthening the nanocomposite and raising the TS. Nevertheless, under stress, the networked structure reduces the elongation of the matrix by preventing molecular movement.

$$\boldsymbol{TS}\ (\text{MPa}) = +11.54 + 0.93A + 0.31B + 0.43C - 0.76AB + 0.63AC + 0.29BC + 0.58A^2 - 0.710B^2 - 0.65C^2 \quad \text{(Equation 3)}$$

$$\boldsymbol{Eb}\ (\%) = +63.52 + 6.74A + 5.26B - 3.46C - 5.34AB + 2.36A^2 + 1.55B^2 \quad \text{(Equation 4)}$$

Where A, B, C are the various coded values of the independent variables.

Table 3: Model summary statistics

Source	Std. Dev.		R-Squared		Adjusted R-Squared		Predicted R-Squared		PRESS		Remark
	TS	*EB*	*TS*	*EB*	*TS*	*EB*	*TS*	*EB*	*TS*	*EB*	
Linear	1.42	4.67	0.3452	0.7541	0.2231	0.7395	-0.1605	0.5682	47.96	751.42	
2FI	1.32	3.35	0.5145	0.9124	0.2785	0.8606	0.0344	0.8144	38.61	276.61	
Quadratic	0.32	1.82	0.9732	0.9584	0.9481	0.9593	0.8237	0.8553	7.42	197.86	Suggested
Cubic	0.22	1.89	0.9932	0.9463	0.9845	0.9463	0.7638	-0.5891	9.43	2626.28	Aliased

Table 4 displays the significant model terms for the answers together with the analysis of variance (ANOVA) for the fitted quadratic model. The model's significance is indicated by its F-values of 51.45 for elongation and 36.25 for tensile strength. The F-value this high caused by noise is about 0.01% and the significant model terms indicated by p-values are less than 0.05. Result show that A, B, C, AB, A2, and B2 are the only important parameters for the elongation at break and all other model terms are significant for tensile strength.

Table 4: ANOVA for response surface quadratic model

Source	**Sum of Squares**		**Df**		**Mean Square**		**F Value**		**p-value**		**Remark**
	TS	*EB*	*TS*	*EB*	*TS*	*EB*	*TS*	*EB*	*TS*	*EB*	
Model	39.64	1647.52	9	9	4.43	173.27	36.25	51.46	< 0.0001	< 0.0001	**Significant**
A-CMC	11.31	636.41	1	1	11.12	642.43	88.67	192.19	< 0.0001	< 0.0001	
B-PECTIN	1.31	393.56	1	1	1.21	394.66	9.64	117.51	0.0111	< 0.0001	
C-CL	1.47	173.80	1	1	1.48	174.82	11.75	51.42	0.0064	< 0.0001	
AB	3.51	228.12	1	1	3.50	238.12	27.90	67.51	0.0004	< 0.0001	
AC	2.97	2.33	1	1	2.87	2.33	23.82	0.68	0.0007	0.4255	
BC	0.66	3.00	1	1	0.66	3.00	5.24	0.78	0.0453	0.3682	
A^2	4.74	80.30	1	1	4.81	80.60	38.33	23.18	0.0001	0.0006	
B^2	7.16	34.42	1	1	7.07		56.41	10.28	< 0.0001	0.0096	
C^2	5.73	0.092	1	1	5.81		46.37	0.027	< 0.0001	0.8721	
Residual	1.25	34.74	10	10	0.13						
Lack of Fit	0.90	23.56	5	5	0.18		2.54	2.31	0.1734	0.1822	**not significant**
Pure Error	0.35	10.22	5	5	0.071						
Cor Total	40.82	1654.37	19	19							

Furthermore, the lack of fit F-value of 2.54 and p-value of 0.17 implies that there is insignificant lack of fit relative to pure error for the tensile strength. The elongation at break also presented an insignificant lack of fit, with an F-value of 2.31 and p-value of 0.18. This indicates that there is 17.48% and 18.65% chance that lack of fit this large could occur due to noise for the tensile strength and elongation at break respectively. This shows that the model is appropriate for the experiment. With values of 0.9732 for tensile strength and 0.9584 for elongation at break, the R^2 is thus quite near to unity. With discrepancies of less than 0.2, the corrected R^2 and the projected R^2 also correspond fairly well. The signal to noise ratio was measured with an adequate precision of 25.355 for tensile strength and 26.45 for elongation at break, both of which are higher than the minimum required ratio of 4.

3.3 Morphological analysis of the nanocomposite films

Figures 2.0 and 3.0, displays scanning electron microscope (SEM) images of cassava lignin in the carboxymethyl cellulose-pectin matrix. The micrograph of the unreinforced film as shown in Figure 2A revealed several discontinuous fissures and loose granules caused by insufficient homogeneity. Furthermore, scanning electron microscopy was used to examine runs 4, 8, and 16 with the highest tensile performance, which had cassava lignin concentrations of 0.75 wt. %, 1.20 wt. %, and 1.65 wt. %. The nanocomposite films inoculated with 0.75 wt.% and 1.20 wt.% of cassava lignin showed improved cohesive structure and interaction between cassava lignin and matrix while 1.65 wt.% cassava lignin reinforced carboxymethyl cellulose-pectin film exhibited some inhomogeneity and deep crack surface as shown in Figures 2B and 3A. This indicates higher cassava lignin fibre reinforcement leading to a decrease in dispersion and more agglomeration. This observation is consistent with the report of Nwanna et al. (2023) [21].

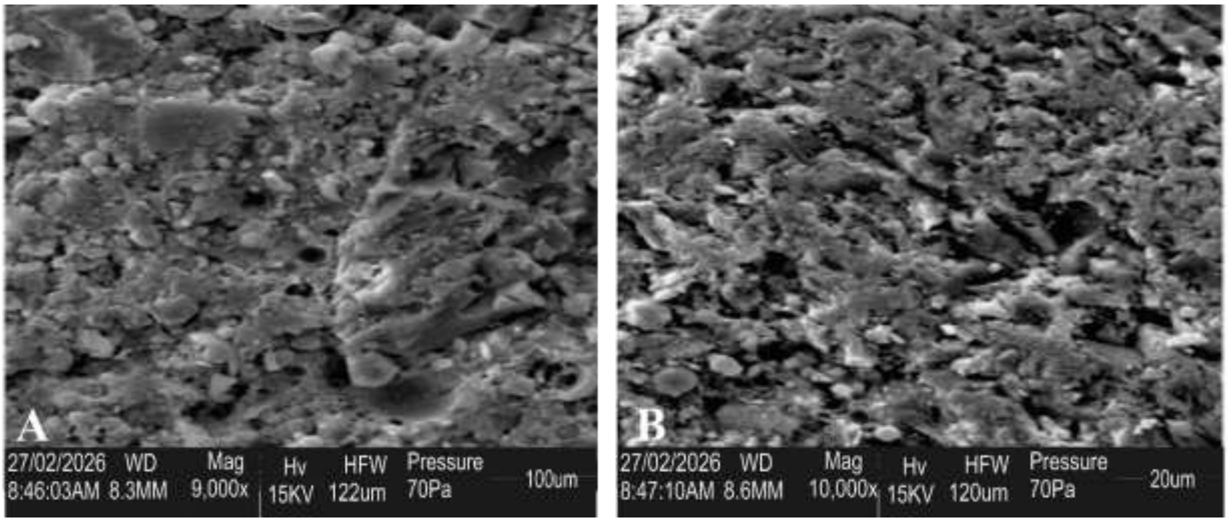

Figure 2.0: SEM image of (a) unreinforced and (b) 0.75 wt.% lignin fibre reinforced films

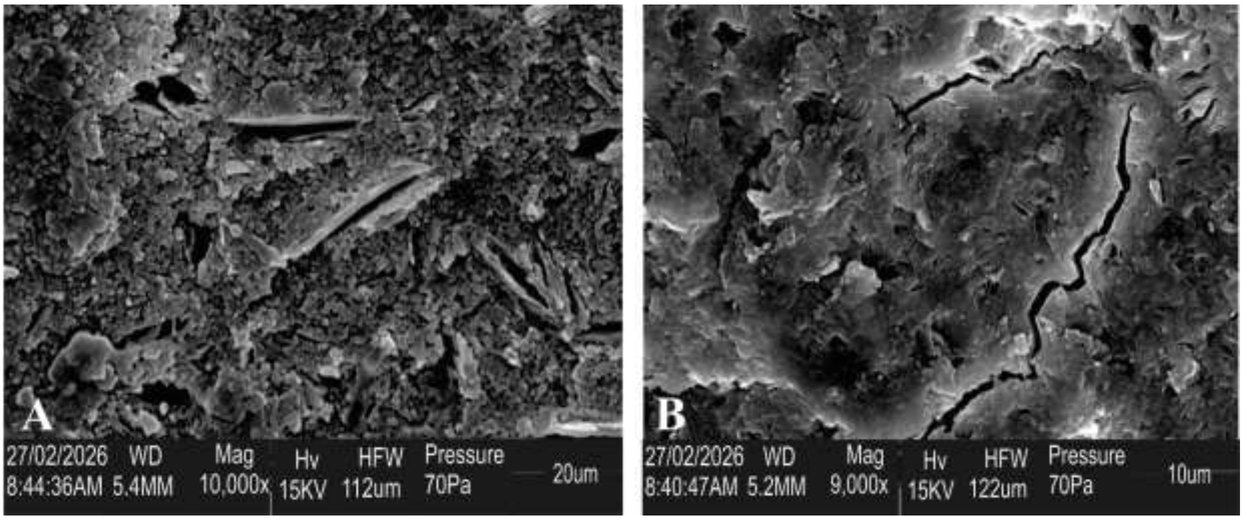

Figure: 3 SEM image of (a) 1.20 wt.% and (b) 1.65 wt.% lignin fibre reinforced films

6. Conclusions

The tensile strength and elongation at break responses studied using the central composite design showed that carboxymethyl cellulose, pectin and cassava lignin all have significant effect on the mechanical properties (tensile strength and elongation at break). The nanocomposite films have strength between 7MPa and 15MPa and the elongation at break between 50% and 100%. The cassava lignin, pectin and carboxymethyl cellulose had effect on the tensile strength while the matrices contributed greatly to the elongation at break. It was established that the 1.65 wt. % cassava lignin reinforced carboxymethyl cellulose-pectin film showed some inhomogeneity and a deep fracture surface, the nanocomposite films inoculated with 0.75 wt. % and 1.20 wt. % of cassava lignin demonstrated enhanced cohesive structure and interaction between cassava lignin and matrix. This suggests that increased cassava lignin fiber reinforcing causes greater agglomeration and less dispersion. The range of tensile strength found in this study is comparable to the values found in other studies [19, 22] and falls within the range that can be taken into consideration for food wrapping applications.

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