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RESEARCH ARTICLE

PHYSICOCHEMICAL CHARACTERIZATION OF NATIVE AND ACETYLATED MUCILAGES FROM SELECTED PLANTS FOR POTENTIAL USE IN ORAL FILM FORMULATIONS

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Abstract

Background: Natural mucilages have gained attention as sustainable polymeric excipients for oral film formulations due to their biodegradability and availability. However, their practical application is limited by high viscosity and excessive swelling, which negatively affect film casting andperformance. The growing demand for patient-centred and patient-specific dosage forms has increased interest in excipients suitable for sublingual drug delivery, a route that offers improved bioavailability by bypassing the first-pass metabolism. Although natural mucilages have been explored as film-forming matrices, their physicochemical limitations necessitate modification. This study aimed to improve the suitability of selected natural mucilages for oral film formulation through acetylation.
Methods: Native mucilages from *Azanza garckeana* fruit, *Abelmoschus esculentus* pods, *Cissus populnea* stem, *Sesamum indicum* leaves, and *Grewia mollis* stem were chemically modified by acetylation, and placebo films prepared by solvent-casting method from both native and acetylated mucilages were evaluated for physicochemical properties.
Result: Fourier transform infrared spectroscopy confirmed successful acetylation indicated by appearance of ester carbonyl stretching bands and reduced hydroxyl stretching intensity. Acetylation significantly reduced viscosity, swelling index and moisture content compared with native mucilages, while pH values remained within acceptable limits for oral administration. Placebo films prepared from some acetylated mucilages, particularly Grewia and Abelmoschus, exhibited physicochemical properties comparable to those of hydroxypropyl methylcellulose.
Conclusion: Acetylation effectively enhanced the functional properties of the studied mucilages, improving their performance as biodegradable film-forming excipients and supporting their potential use in oral film formulations.
Keywords: Acetylation, mucilage, natural polymers, oral films, physicochemical properties.

INTRODUCTION

Oral dosage forms are widely used because of their convenience and patient acceptability; however, conventional tablets and capsules are often unsuitable for paediatric and geriatric patients, individuals with dysphagia, and situations requiring rapid onset of action[1]. Oral disintegrating films (ODFs) have emerged as an effective alternative, as they are thin, flexible polymeric matrices that disintegrate rapidly in the oral cavity without the need for water, thereby improving patient compliance and therapeutic response[2]. ODFs are generally required to disintegrate within 30 seconds (for immediate release films), few minutes (for mucoadhesive meltaway films) and between 8 to 10 hours (for mucoadhesive sustained release films), while maintaining sufficient mechanical strength for handling, packaging, and transport[3].

ODFs are particularly suited for buccal and sublingual administration, where partial or complete avoidance of first-pass metabolism can enhance bioavailability. Their rapid disintegration and ease of administration make them especially valuable for acute therapeutic interventions and vulnerable populations. The performance of ODFs is strongly influenced by the properties of the film-forming polymer, which constitutes between 40 to 65 % of the total dry weight of the film[3]. Natural gums and mucilages are gaining

interest as biodegradable and locally sourced excipients, but their high viscosity, uncontrolled swelling, and variable film quality limit direct application in ODF formulations[4].

Viscosity and swelling behaviour determine film castability and disintegration kinetics. Excessive viscosity leads to poor film uniformity and processing challenges, while high swelling can delay saliva penetration and extend disintegration times beyond acceptable limits[2,5]. These limitations necessitate polymer modification to tailor hydration and mechanical behaviour for rapid disintegration films. Chemical modification, such as acetylation, can reduce hydrophilicity, modulate hydrogen bonding, and decrease polymer chain entanglement, leading to lower viscosity, controlled water uptake, improved castability, and enhanced film integrity[6].

This study therefore aimed to modify selected native mucilages by acetylation and to evaluate their viscosities and film-forming capacities as matrices for oral disintegrating film formulations.

MATERIALS AND METHODS

Plant materials were collected and authenticated at Gombe State University, Gombe, Nigeria and assigned voucher numbers GSUH 82, GSUH 198, GSUH 236, GSUH 298, GSUH 368 for *Cissus populnea*, *Azanza garckeana*, *Abelmoschus esculentus*, *Sesamum indicum* and *Grewia mollis* respectively. Salbutamol sulfate powder, distilled water, citric acid (PS PARK Scientific limited, England), anhydrous acetic anhydride (Surechem Product)**,** sodiums hydroxide, sodium metabisulfite (all chemicals used are of analytical grade), pH meter (Setra model no: PHS-25, Germany).

Extraction of mucilages

Mucilages were extracted using established methods. *A. garckeana* mucilage was extracted as described by Belkheiri *et al.*[7], *A. esculentus* according to Nagpal *et al.*[8], while *C. populnea* and *G. mollis* mucilages were obtained following Adeleye *et al.*[9], *S. indicum* mucilage was extracted using the method reported by Ngwuluka *et al*[10]. The dried mucilages were pulverized, passed through a 180 µm sieve, and stored in airtight containers.

Acetylation of native mucilages

Ten (10) grams of each mucilage were dispersed in distilled water and alkalized to pH 8. Acetic anhydride was added and the reaction mixture maintained at 45°C for 90 minutes. The product was washed to neutrality, dried, pulverized, and sieved as previously described[11].

Organoleptic and physicochemical evaluation of native and acetylated mucilages

Organoleptic evaluation was done by sensory assessment of the mucilages using human senses to characterize the colour, odour, texture and taste of the mucilages. Moisture content and swelling index were determined using the method of Emenike *et al.*[12]. The pH of 1 % w/v mucilage dispersions was measured using a calibrated digital pH meter. Viscosity of 1 % w/v solutions was determined using an Ostwald viscometer[10]. FTIR spectroscopy was employed to confirm chemical modification.

Preparation of oral films

Oral films were prepared by solvent casting at using 40 % w/w, 50 % w/w and 60 % w/w (1.33, 1.67 and 2 % w/v respectively) of both native and acetylated mucilages with glycerolas plasticizer (Table 1). The mucilages were separately dispersed in 30 mL of distilled water and allowed to hydrate for few hours. Glycerol was then added, stirred properly and kept aside for few minutes to allow the bubbles to settle. The dispersions were poured into petri dishes and dried at 60°C in a hot air oven. Dried films were carefully cut into squares (2x2 cm^2) and selected based on peelability, smoothness and homogeneity, and then evaluated for film properties including appearance, thickness, weight uniformity, folding endurance, surface pH, swelling index and disintegration time using standard procedures[13].

Table 1: Formulation design of placebo films using native and acetylated mucilages.

Component/Formulation	1	2	3
G (g)	0.4	0.5	0.6
% w/v	1.33	1.67	2.0
Glycerol (mL)	0.5	0.5	0.5
Distilled Water (mL)	30	30	30

RESULTS AND DISCUSSION

The result for the yield, organoleptic and physicochemical properties of the mucilages are shown in Table 2 and Table 3. The yield (%) of the extracted mucilages was generally low, which is characteristic of natural gums. Variations in yield among the plant sources may be attributed to differences in botanical origin, harvesting conditions, and extraction parameters such as temperature, time, pH, and solvent system. Similar observations have been reported for *C. populnea*, *G. mollis*, and other plant mucilages, where extraction variables significantly influenced yield and functional performance[14,15]. These findings confirm that mucilage yield is largely dependent on polysaccharide content and plant part used rather than extraction efficiency alone.

Organoleptic evaluation showed that all mucilages were brown to off-white, bland in taste, and largely odourless, except for *A. esculentus* and *C. populnea*, which exhibited characteristic pleasant odours. These properties are consistent with previous reports on natural mucilages and are typical of polysaccharide-rich materials containing associated phytoconstituents such as polyphenols and flavonoids[14]. Viscosity is a critical parameter in selecting polymers for oral disintegrating films, as it influences film thickness, mechanical strength, and disintegration behavior. Native mucilages exhibited significantly higher viscosities than their acetylated counterparts ($p<0.001$), reflecting extensive intermolecular hydrogen bonding within the polysaccharide chains. Statistical analysis revealed that *Sesamum indicum* mucilage was comparable to HPMC, indicating its potential suitability as a viscosity-dependent film former.

Table 2: Yield and organoleptic properties of the mucilages.

Mucilages	% Yield	Colour	Odour	Texture	Taste
A. garckeana	2.70	Brown	Odourless	Gritty	Bland
A. esculentus	1.12	Brown	Characteristic	Gritty	Bland
G. mollis	4.02	Brown	Odourless	Gritty	Bland
C. populnea	3.73	Brown	Pleasant	Powdery	Bland
S. indicum	1.24	Brown	Odourless	Gritty	Bland
HPMC	-	Off-white	Odourless	Powdery	Bland

Table 3: Physicochemical properties of the mucilages.

Mucilages	pH	Viscosity (m^2/s)	Swelling index	% Moisture content
Azanza (Native)	7.00	289.88	10.78	10.60
Acetylated	6.40	276.65	10.33	10.42
Abelmoschus (Native)	6.51	312.45	4.60	12.80
Acetylated	6.31	299.83	4.50	11.15
Grewia (Native)	7.47	320.37	8.27	10.72
Acetylated	7.21	299.75	8.18	10.02
Cissus (Native)	6.78	249.68	10.33	10.48
Acetylated	6.15	215.42	3.73	10.11
Sesamum (Native)	7.47	331.12	3.33	10.40
Acetylated	7.10	300.01	3.11	10.15
HPMC	7.50	300.16	6.19	8.20

Viscosity directly influences film-forming ability, mechanical strength, and drug release behaviour in oral film formulations[10-12]. In contrast, *C. populnea* showed significantly lower viscosity, suggesting weaker chain entanglement or lower molecular weight. Acetylation markedly reduced viscosity, swelling index, and moisture content across all mucilages. This reduction is attributable to substitution of hydrophilic hydroxyl groups with bulkier, less polar acetyl groups, which disrupt hydrogen bonding, reduce hydration capacity, and promote a more compact polymer conformation in solution[7]. From a formulation perspective, this modification is advantageous, as excessively high viscosity can hinder water penetration and delay film disintegration[15,16]. Swelling index followed a similar trend, with native mucilages exhibiting higher swelling due to the abundance of free hydroxyl groups available for water binding. Acetylation reduced swelling by decreasing polymer hydrophilicity and altering network porosity, consistent with reports on chemically modified poly-saccharides[15,17]. Controlled swelling is desirable in oral films to balance rapid hydration with structural integrity. The pH values of both native and acetylated mucilages were within acceptable limits for oral administration, indicating minimal risk of mucosal irritation. Slightly lower pH values observed in acetylated mucilages are consistent with esterification and do not compromise suitability for oral use[3]. Moisture content significantly influences the mechanical and stability properties of films. The acetylated mucilages exhibited lower moisture than the native forms, reducing the risk of excessive plasticization, microbial growth, and processing challenges.

Table 4: Mean comparisons of viscosities of native mucilage using two-way ANOVA.

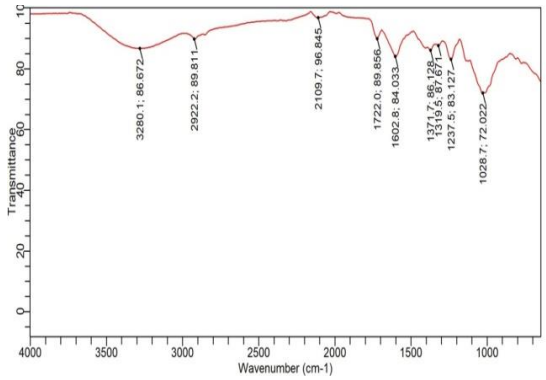
Viscosity	Sum of Square	Df	Mean Square	F	Sig.
Between groups	17218.434	5	3443.687	21359.141	0.000
Within groups	1.935	12	0.161		
Total	17220.369	17			

Table 5: Post Hoc Test (Turkey) to determine the variability in viscosities of the mucilages.

Mucilage properties	Comparisons	*p*-values
AZ	A	0.001*
	G	0.001*
	C	0.001*
	S	0.001*
	HPMC	0.001*
A	G	0.708
	C	0.001*
	S	0.001*
	HPMC	0.993
G	C	0.001*
	S	0.645
	HPMC	0.404
C	S	0.001*
	HPMC	0.001*
S	HPMC	0.997

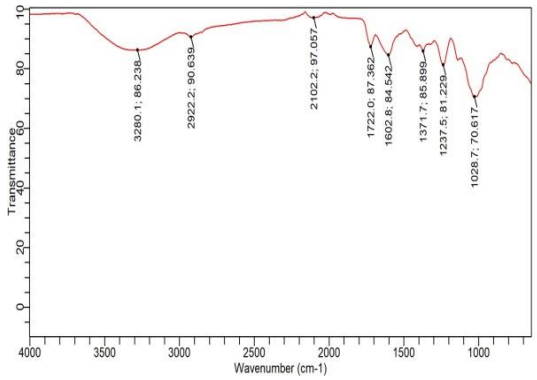
Table 6: Physicochemical and mechanical properties of the films.

Formulations	Weight (g)	Thickness (mm)	Folding endurance	% Elongation	pH	Swelling index	Disintegration time (min.)
N-Ae$_{50}$	0.118	0.190	>300	20	7.14	5.97	15.43
N-Ae$_{60}$	0.127	0.160	>300	15	6.74	5.04	37.20
A-Ae$_{50}$	0.055	0.130	>300	20	6.97	4.94	11.43
A-Ae$_{60}$	0.077	0.150	>300	15	6.58	4.72	13.67
N- G$_{50}$	0.052	0.130	>300	10	6.82	6.77	19.32
N-G$_{60}$	0.080	0.150	>300	10	6.20	6.42	23.25
A-G$_{50}$	0.052	0.130	>300	15	6.91	2.59	13.20
A-G$_{60}$	0.080	0.160	>300	10	6.80	2.42	17.32
A-Az$_{50}$	0.055	0.120	>300	25	6.71	7.17	7.46
A- Az$_{60}$	0.053	0.130	>300	20	6.02	4.89	11.31
N- S$_{60}$	0.097	0.100	>300	40	6.73	5.92	25.40
A- S$_{50}$	0.048	0.120	>300	55	6.44	1.34	29.13
A-S$_{60}$	0.099	0.150	>300	50	6.23	1.22	31.28
H$_{40}$	0.047	0.130	>300	10	7.20	5.35	4.02
H$_{50}$	0.069	0.160	>300	10	6.99	4.85	15.38
H$_{60}$	0.090	0.190	>300	12	6.72	4.71	27.03



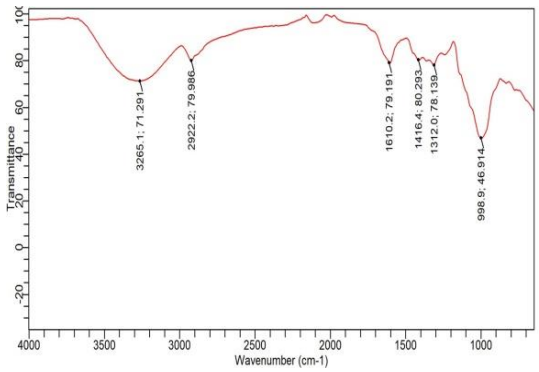
Peak Number	Wavenumber (cm^{-1})	Intensity
1	1028.74524	72.02201
2	1237.47616	83.12742
3	1319.47759	87.67108
4	1371.66032	86.12776
5	1602.75527	84.03334

Figure 1: FTIR spectrum of native *A. garckeana* mucilage.



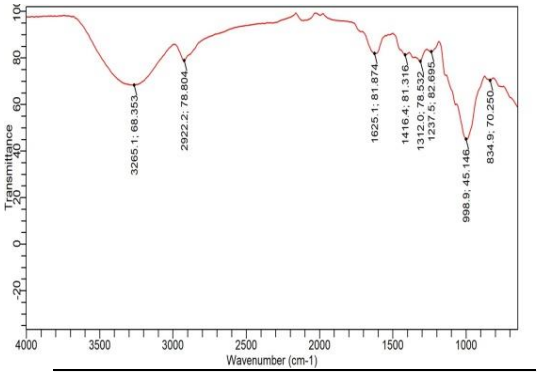
Peak Number	Wavenumber (cm^{-1})	Intensity
1	1028.74524	70.61662
2	1237.47616	81.22934
3	1371.66032	85.89950
4	1602.75527	84.54156
5	1722.03008	87.36179

Figure 2: FTIR spectrum of acetylated *A. garckeana* mucilage.



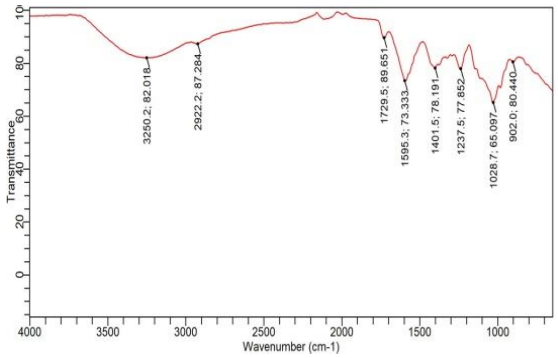
Peak Number	Wavenumber (cm^{-1})	Intensity
1	998.92654	46.91383
2	1312.02292	78.13933
3	1416.38838	80.29307
4	1610.20995	79.19116
5	2922.23286	79.98551

Figure 3: FTIR spectrum of native *C. populnea* mucilage.



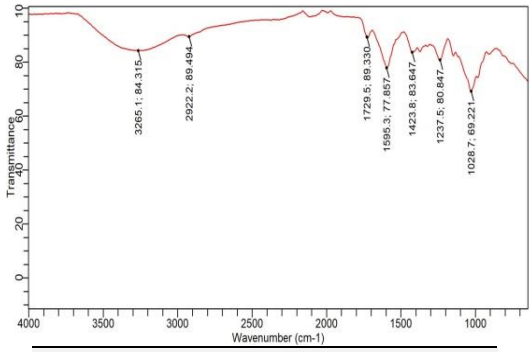
Peak Number	Wavenumber (cm^{-1})	Intensity
1	834.92368	70.25013
2	998.92654	45.14575
3	1237.47616	82.69516
4	1312.02292	78.53184
5	1416.38838	81.31591

Figure 4: FTIR spectrum of acetylated *C. populnea* mucilage.



Peak Number	Wavenumber (cm^{-1})	Intensity
1	902.01576	80.43969
2	1028.74524	65.09672
3	1237.47616	77.85152
4	1401.47903	78.19130
5	1595.30059	73.33328

Figure 5: FTIR spectrum of native *G. mollis* mucilage.



Peak Number	Wavenumber (cm^{-1})	Intensity
1	1028.74524	69.22074
2	1237.47616	80.84689
3	1423.84305	83.64714
4	1595.30059	77.85732
5	1729.48476	89.33009

Figure 6: FTIR spectrum of acetylated *G. mollis* mucilage.

Most values were within the recommended limits for pharmaceutical polymers, supporting their suitability for film formulation[15].

Fourier Transform Infrared (FTIR) analysis

FTIR spectra of native and acetylated mucilages (Figure 1 to Figure 6) confirmed successful chemical modification. Native samples exhibited characteristic polysaccharide bands, including broad O–H stretching, C–H stretching, and C–O–C vibrations. Acetylated samples showed the appearance of ester-related C–O stretching bands in the 1230–1245 cm^{-1} region and reduced O–H band intensity, indicating substitution of hydroxyl groups by acetyl moieties.

Minor shifts in hydrogen-bond-related regions further supported structural modification. These spectral changes collectively confirm successful acetylation while preserving the polysaccharide backbone.

Preliminary properties of placebo films

Table 6 below shows the physicochemical properties of the films formulated with both native and acetylates mucilages compared with HPMC as standard. All the mucilages with the exception of HPMC formulated with 0.4g (1.33% w/v) were very sticky, not peelable and could not be used for further evaluation. The films formulated with both native and acetylated *C. populnea* mucilage and native *A. garckeana* at both 0.5g (1.67 % w/v) and 0.6g (2% w/v) and native *S. indicum* at 0.5 g were not peelable, had poor handling and could not undergo further evaluation, while those of native and acetylated *G. mollis, A. esculentus* at 0.5g (1.67% w/v) and 0.6g (2% w/v), native *S. indicum* mucilages at 0.6g (2% w/v), acetylated *S. indicum* at 0.5g and 0.6g (2% w/v) and acetylated *A. garckeana* at 0.5g (1.67% w/v) and 0.6g (2% w/v) were peelable and had good film properties. The physicochemical and mechanical properties of the placebo oral films formulated from native and acetylated mucilages are summarized in Table 6. These parameters are critical determinants of film processability, stability, patient acceptability, and in-use performance. Film weight and thickness were relatively uniform across formulations, indicating good reproducibility of the solvent casting process.

The slightly higher weights and thicknesses observed at increased polymer concentrations (60% w/v equivalents) are expected and reflect greater solid content in the casting solution. Uniform thickness is essential for dose accuracy and consistent disintegration behavior in oral disintegrating films (ODFs).

All films exhibited folding endurance values greater than 300, demonstrating excellent mechanical flexibility and resistance to cracking. This indicates adequate polymer chain entanglement and plasticization, which are necessary for handling, packaging, and transport. Importantly, acetylation did not compromise film flexibility, suggesting that reduced hydrogen bonding did not adversely affect mechanical integrity.

Percent elongation values varied among formulations and generally decreased with acetylation. This reduction is attributable to decreased intermolecular hydrogen bonding and lower chain cohesion following acetyl substitution of hydroxyl groups. While native mucilages showed higher elongation due to extensive hydration and gel formation, acetylated films exhibited a more controlled elastic response, which is desirable for maintaining film shape during handling without excessive deformation. Percent elongation describes the strength of films under tensile stress. An ODF should have a percent elongation of 0.3-38 % as reported in literature[3]. It was observed that as the concentration of the mucilage increased, the percent elongation decreased, the concentration of polymer used and amount of plasticizer affects the percent elongation of films.

Surface pH values for all films remained within the near-neutral range (6.0–7.2), which is considered safe and non-irritating for the oral mucosa. This confirms the suitability of both native and acetylated mucilages for buccal and sublingual application and indicates that acetylation did not introduce acidity or alkalinity that could compromise patient comfort. Swelling index showed a marked reduction in acetylated films compared with native counterparts. Native mucilages

exhibited higher swelling due to the abundance of hydrophilic hydroxyl groups that promote rapid water uptake and gel layer formation. Acetylation reduced polymer hydrophilicity and intermolecular hydrogen bonding, thereby limiting excessive swelling. Controlled swelling is essential in ODF systems, as excessive gel formation can delay saliva penetration and prolong disintegration time.

Disintegration time is a critical performance parameter for ODFs. Thickness and weight of the films play a significant role in determining the disintegration time of water-soluble films[3]. Disintegration is a battle between hydration and cohesion. Acetylated mucilage films generally showed faster and more controlled disintegration compared with native films. The reduced swelling and lower viscosity associated with acetylation facilitated rapid saliva ingress and matrix breakup of the films. In contrast, native mucilages, particularly at higher concentrations, exhibited prolonged disintegration due to dense gel barrier formation. The performance of acetylated Grewia and Abelmoschus films was comparable to hydroxypropyl methylcellulose (HPMC), highlighting their potential as natural alternatives to synthetic film-forming polymers. Although these formulations exceeded the ideal ≤30 seconds disintegration threshold, the observed improvements demonstrate that acetylation significantly enhances the functional suitability of natural mucilages for ODF applications. Disintegration time of the oral films can be enhanced through the addition of a super-disintegrant especially when formulating flash release oral disintegrating films[17].

The result shows that chemical modification of mucilages by acetylation reduces polymer chain interactions and lowers solution viscosity compared with the native mucilage and, in polymer film systems, this reduced viscosity facilitates better dispersion and casting uniformity, leading to improved physical properties including optimal moisture content, controlled swelling behavior, uniform pH and appearance, and faster disintegration times of the films. The native mucilages when compared with the acetylated ones have shown to produce films with distinctly different mechanical and functional properties, with the acetylated derivatives producing films with superior processing and performance characteristics compared with the native.

Limitations of the study

Successful acetylation of the mucilages was only inferred from FTIR, degree of acetylation/substitution was not determined. Only placebo films (films without an active pharmaceutical ingredient) were formulated and used to compare the mechanical properties of the native and acetylated mucilages.

CONCLUSION

Acetylation effectively modified the physicochemical properties of mucilages from the selected plants, improving their functional suitability as film-forming excipients. The acetylated mucilages demonstrated properties comparable to HPMC, supporting their potential application in biodegradable oral film formulations.

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AUTHOR'S CONTRIBUTION

Audu-Peter JD: conceived the study, supervised the experimental work. **Samaila AB:** laboratory experiments, data analysis. **Ngwuluka NC:** study design, data interpretation, revision. Final manuscript was checked and approved by all authors.

DATA AVAILABILITY

The empirical data used to support the study's conclusions are available upon request from the corresponding author.

CONFLICT OF INTEREST

No conflict of interest associated with this work.

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