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Preparation of Conductive Antibacterial Film of Organoclay Origin

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Abstract: *This study presents the synthesis and characterization of novel conductive antibacterial thin films derived from Kashikoko and Kaffin-Koro biopolymers, modified with phenylamine and silver nitrate. The average thickness of the films was measured to be 0.33 mm for Kashikoko/PA, 0.40 mm for Kashikoko/CMC/EG, and 0.30 mm for Kaffin-Koro/PA, demonstrating their structural integrity. Electrical conductivity measurements revealed that the Kashikoko/PA/0.3 sample exhibited the highest conductivity at 1818 S/cm, while the Kashikoko/PA/1.0 sample recorded a lower conductivity of 950 S/cm. Antimicrobial efficacy was assessed by measuring the inhibition zones against E. coli, Salmonella, and S. aureus. The results showed that the Kashikoko/CMC/EG/PA/1g/AgNO3 exhibited the largest inhibition zone of 34 mm against E. coli. At the same time, the Kaffin-Koro/CMC/EG/PA/0.2/$AgNO_3$ displayed a zone of inhibition ranging from 19-36 mm across all tested pathogens, with the highest activity against S. aureus (36 mm). These findings indicate that the developed nanocomposite films possess significant electrical conductivity and antimicrobial properties, making them suitable for applications in active food packaging and biomedical fields.*

Keywords*: Conductive films, antibacterial films, biopolymer films, silver nitrate, antimicrobial efficacy, food packaging*

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1.0 Introduction

The increasing demand for materials that exhibit both electrical conductivity and antibacterial properties has prompted significant research into novel film formulations. Conductive antibacterial films have applications across various fields, including packaging, electronics, and healthcare, where they can mitigate bacterial growth and enhance product performance (Motelica *et al.*, 2020; Fadiji *et al.*, 2023; Bahmid *et al.*, 2024; Nithya *et al.*, 2023). Among the promising materials for developing these films is organoclay, which combines the unique properties of clay minerals with organic modifications to enhance their dispersion, compatibility, and performance in polymer matrices (Khan *et al.*, 2023). Organoclays have been widely studied for their potential to create multifunctional materials due to their high surface area, mechanical strength, and inherent antibacterial properties (Shaheen *et al.* 2019; Zhang *et al.*, 2023). However, despite the growing body of research on organoclay-based composites, there remains a significant knowledge gap in understanding the precise mechanisms by which these materials can impart conductivity and antibacterial properties simultaneously. While some studies have explored the synthesis of conductive films using various additives, few have focused specifically on organoclay as a primary component for these functionalities (Abdul Rahman *et al.*, 2023). Also, the optimization of the film preparation process to achieve the desired balance of conductivity, mechanical properties, and antibacterial efficacy has not been comprehensively addressed in the literature.

This study aims to fill this gap by investigating the preparation of conductive antibacterial films derived from organoclay. The specific objectives of this research include (1) the synthesis of organoclay composites incorporating conductive fillers, (2) the characterization of their physical, mechanical, and electrical properties, and (3) the evaluation of their antibacterial activity against common pathogens. Through this work, we aim to

develop a comprehensive understanding of the potential of organoclay as a versatile material for producing functional films that meet the demands of modern applications.

2.0 Materials and Methods

2.1 Materials

Carboxyl methyl cellulose, Arginine ($C_6H_5NH_2$) with phenylalanine, Hydrogen Peroxide (H_2O_2), Sodium Chloride (NaCl), Hydrochloric Acid (HCl), Acetic acid, ethylene glycol, 0.1% NaOH, 0.1% $AgNO_3$, Nutrient agar, Nutrient broth, Dimethyl sulfur oxide (DMSO), salt and Distilled water. All reagents were of analytical grade and were used as received.\

2.2 Methods

2.2.1 Sampling and Sample Treatment

The sample used in this project research was a Nanoclay sample synthesized from the native clay collected at Kaffin-Koro and Kashikoko areas in Paikoro and Bida Local Government Areas respectively.

2.2.2 Preparation of Nanoclay

The synthesis of nano clay was carried out using the method of Elele *et al.* (2020) and Azeh *et al.* (2021). In a typical experiment, 1 kg fine powdered native clay was suspended in 5 L of distilled water and vigorously stirred followed by adding 100 mL of hydrogen peroxide. The content was allowed to stand for 72 h to settle. Repeated sedimentation and decantation were carried out to prepare Nanoclay. Hydrogen peroxide was introduced to degrade the organic matter into water and carbon (IV) oxide. The clay suspension was saturated with NaCl to enrich the clay surface with sodium ions.

$$\text{Clay-Organic matter} + H_2O_2\,(l) \longrightarrow CO_2\,(g) + H_2O\,(l) \longrightarrow \text{Clay-}M^{n+}\,(aq) + NaCl\,(aq) \longrightarrow \text{Clay-}Na^{+}\,(s) + M^{n+}Cl\,(aq) \longrightarrow \text{clay-anion}$$

2.2.3 Phenylalanine and arginine incorporation in nano clay

300 mL of the Nano clay suspension was measured and transferred into a 600 mL beaker followed by the addition of 300 mL of phenylalanine solution was added and stirred for 2 h for the both Kashikoko and Kaffin-Koro Nano clay samples. The same procedure was used for 250 mL of arginine. The modified Nanoclay was then, filtered and dried. It was pounded and sieved using a standard sieve.

2.2.4 Silver nitrate ($AgNO_3$) incorporation in nano clay

Five grams (5 g) of Kashikoko modified with phenylalanine was weighed in a beaker, 20 mL of 0.1 % $AgNO_3$ was added to the sample and stirred, it was poured into a petri dish and dried under the sun.

2.2.5 Preparation of the films

One gram (1.00 g) of CMC was weighed in a 100 mL beaker, 10 mL of 0.1 % NaOH was added and stirred, 20 mL of water was added and stirred continuously, 10 mL of 0.1 % NaOH was added, 10 mL of 10 % acetic acid was added and constantly stirred, another 10 mL of 0.1% NaOH was added again and stirred continuously, 2.5 mL of ethylene glycol was added and constantly stirred until there were no more lumps, 0.5 g of Kashikoko clay was suspended with small amount of water and added, it was stirred continuously for 2 h (Azeh *et al.*, 2023). The suspension was cast in a petri dish and glass plate.

2.3 Thickness of film

The thickness of the film was measured using a digital calliper. The thickness was measured at three different portions of the film. The result was noted and the average was calculated.

2.4 Electrical conductivity of film

The electrical conductivity of the films was measured using a conductivity meter. The weight of the film was first determined using a weighing balance to know the amount of distilled water to be added to swell the film. Each film was inserted into a test tube and diluted with distilled water according to the weight of the film, it was allowed to stand for 30 min at room temperature and the electrical conductivity was taken. The electrical

conductivity of distilled water was also taken (Azeh *et al.*, 2023). The electrical conductivity of the distilled water was subtracted from that of the films.

2.5 Antibacterial analysis

2.5.1 Preparation of medium

A nonsynthetic medium was used. Nutrient agar was prepared by dissolving 28.0 g of the powdered medium in a litre of distilled water. It was autoclaved(sterilized) at 121 ^{o}C for 15 min and at a pressure of 1.5 Nm^{-2}. The sterilized medium was allowed to attain dispersing temperature (45 ^{o}C – 50 °C) and then dispersed into sterile Petri dishes and allowed to solidify before holes were made inside using a sterile cork borer of 6.0 mm in diameter.

The nutrient broth was also prepared by dissolving 13.0 g of the powder into a litre of distilled water.10.0 mL of the broth was dispersed into a test tube and was sterilized under the same condition as the nutrient agar. It was also allowed to cool before usage.

2.5.2 Reconstitution

The biofilm was reconstituted to liquid form by weighing 0.3 g (300 mg) and dissolved in 5.0 mL of Dimethyl sulfur oxide (DMSO). This was further diluted with 3.0 mL of sterile distilled water. Therefore, the concentration of each biofilm is 300 mg/8 mL (37.50 mg/mL). This served as diluents used for the antibacterial activity.

2.5.3 Inoculation

The test bacterium was inoculated into a broth medium (nutrient broth) and was incubated for 30 min at 37 °C. The broth was subcultured into another free sterile broth using a loopful of the wire loop. The subculture broth was incubated for 3 h at 37 °C. The turbidity of the incubated subculture was compared with McFarland's turbidity standard before usage (Azeh *et al.*, 2023). The culture was inoculated into nutrient agar Petri dishes after it had attained the turbidity standard using sterile swab sticks.

2.5.4 Dispersing the liquid biofilm

After the holes in the plates had been inoculated with the test bacteria, 2 mL of the biofilm was introduced into the inoculated and allowed to stand for 2 h for effective diffusion into the wells.

2.5.5 Incubation

The plates containing the test bacteria and diffused biofilm were incubated at 37 ^{o}C for 24 hours.

2.6 Meat analysis

The meat was bought from the market and was used for bacterial analysis. The meat was tested for the presence of microbes before and after the analysis. The meat was wrapped with the antimicrobial film and incubated for three days.

2.7 Fourier transform infrared analysis of synthesized nanocomposites

The FT-IR spectra of nanocomposites were determined using FT-IR-8400S Fourier Transform Infrared Spectrophotometer in the wavelength range of 4000-400 cm^{-1}. Nanocomposite samples were run as (Kbr) pellets.

2.8 Thermogravimetric analysis (TGA)

The thermal degradation profile of the nanocomposites under the control temperature program was analyzed using the Shimadzu TGA 50H (Kyoto, Japan) which operated between 100 ^{o}C to 900 ^{o}C with a heating rate of 10 ^{o}C / min, nitrogen atmosphere and at a flow rate of 20 mL/min.

2.9 Scanning electron microscope (SEM)

The sample morphology was examined using a PhenomWorld ProX desktop scanning electron microscope with a fully integrated EDS detector, in Eindhoven Netherlands.

3.0 Results and Discussion

3.0 Thickness of the Film

The thickness of the film was measured using a digital calliper. Measurements were made at nine random locations of each preconditioned film and the values were reported. The readings

were used to calculate the average **(Table 1)**. Kashikoko modified/PA/0.2/CMC/EG with an average thickness of 0.33, 0.40, and 0.30 mm respectively had good electrical conductivity values of 1818 S/cm, and the diameter zone of inhibition of 27.00, 32.00, and 28.00 mm against *E. coli, salmonella, and S. aureus*.

Table 1: Thickness of the Thin Film Nanocomposites

S/No.	Sample (g)	Reading (mm)					
Kashikoko (PA)		**1**	**2**	**3**	**AV1**	**AV2**	**AV3**
1	1.0	0.7,0.2,0.3	0.7,0.4,0.3	0.3,0.1,0.6	0.4	0.46	0.33
2	0.5	0.3,0.2,0.1	0.1,0.1,0.2	0.5,0.3,0.4	0.2	0.13	0.4
3	0.4	0.2,0.3,0.5	0.2,0.5,0.2	0.4,0.2,0.1	0.33	0.3	0.23
4	0.3	0.4,0.3,0.3	0.5,0.3,0.4	0.2,0.3,0.4	0.33	0.4	0.3
5	0.2	0.6,0.4,0.2	0.2,0.2,0.6	0.3,0.2,0.3	0.4	0.33	0.26
6	0.1	0.1,0.1,0.1	0.2,0.1,0.1	0.1,0.1,0.1	0.1	0.13	0.1
Kashikoko							
1	1.0	0.1,0.2,0.2	0.3,0.2,0.8	0.1,0.8,0.4	0.16	0.43	0.43
2	0.5	0.5,1.6,0.2	0.5,0.7,0.3	0.2,0.3,0.2	0.76	0.5	0.23
3	0.4	0.2,0.1,0.2	0.3,0.5,0.7	0.3,0.3,0.3	0.16	0.5	0.3
4	0.3	0.1,0.1,0.1	0.8,0.2,0.2	0.1,0.1,0.1	0.1	0.4	0.1
5	0.2	0.3,0.2,0.3	0.1,0.1,0.1	0.1,0.1,0.3	0.26	0.1	0.16
6	0.1	0.1,0.1,0.1	0.1,0.1,0.1	0.1,0.1,0.1	0.1	0.1	0.1
Kaffin-Koro (PA)							
1	1.0	0.1,0.1,0.1	0.1,0.1,0.1	0.1,0.1,0.1	0.1	0.1	0.1
2	0.5	0.3,0.2,0.2	0.2,0.3,0.2	0.3,0.2,0.3	0.23	0.23	0.26
3	0.4	0.1,0.1,0.2	0.2,0.4,0.2	0.3,0.2,0.2	0.13	0.26	0.23
4	0.3	0.2,0.2,0.1	0.2,0.1,0.1	0.2,0.2,0.1	0.16	0.13	0.16
5	0.2	0.1,0.6,0.2	0.1,0.1,0.2	0.1,0.2,0.1	0.3	0.13	0.13
6	0.1	0.1,0.1,0.1	0.1,0.1,0.2	0.1,0.2,0.1	0.1	0.13	0.13
Kaffin-Koro (Ar)							
1	1.0	0.1,0.1,0.1	0.1,0.1,0.1	0.1,0.1,0.1	0.1	0.1	0.1
2	0.5	0.1,0.1,0.1	0.1,0.1,0.1	0.1,0.1,0.1	0.1	0,1	0.1
3	0.4	0.2,0.1,0.2	0.1,0.1,0.2	0.2,0.1,0.1	0.16	0.13	0.13
4	0.3	0.1,0.2,0.2	0.1,0.2,0.1	0.2,0.2,0.2	0.16	0.13	0.2
5	0.2	0.1,0.1,0.1	0.2,0.2,0.2	0.2,0.1,0.2	0.1	0.2	0.16
6	0.1	0.1,0.1,0.1	0.1,0.1,0.1	0.2,0.2,0.2	0.1	0.1	0.2
Kashikoko (C)							
1	1.0	0.2,0.1,0.1	0.1,0.1,0.1	0.1,0.1,0.1	0.13	0.1	0.1
2	0.5	0.1,0.1,0.1	0.1,0.1,0.5	0.1,0.1,0.5	0.1	0.23	0.23
3	0.4	0.1,0.1,0.1	0.1, 0.1, 0.1	0.1, 0.1, 0.1	0.1	0.1	0.1
4	0.3	0.1,0.1,0.1	0.1,0.1,0.1	0.1,0.1,0.1	0.1	0.1	0.1
5	0.2	0.2,0.2,0.1	0.1,0.1,0.1	0.1,0.3,0.2	0.16	0.1	0.2
6	0.1	0.3,0.4,0.1	0.1,0.2,0.1	0.5,0.3,0.1	0.26	0.13	0.3
Kaffin-Koro (C)							
1	1.0	0.1,0.1,0.1	0.1,0.1,0.1	0.1,0.1,0.1	0.1	0.1	0.1
2	0.5	0.1,0.1,0.1	0.1,0.1,0.1	0.1,0.1,0.1	0.1	0.1	0.1
3	0.4	0.1,0.2,0.1	0.1,0.1,0.1	0.2,0.1,0.1	0.13	0.1	0.13

4	0.3	0.1,0.1,0.1	0.3,0.1,0.1	0.3,0.1,0.1	0.1	0.16	0.16
5	0.1	0.1,0.1,0.1	0.1,0.1,0.1	0.1,0.1,0.1	0.1	0.1	0.1

Key: AV1 = Average One; AV2 = Average Two; AV3 = Average Three

3.1 UV-Lamp Spectroscopy of Film

The film was viewed under an ultraviolet lamp at 240 nm. The hybrid film (**Plate I**) showed a bright blue fluorescence, which was attributed to the electronic excitations and transition in the thin film, due to the electronic transition of the non-bonding (Lone-pair) electrons on N- or O-atoms from the non-bonding orbitals to the pi anti-bonding and sigma anti-bonding orbitals respectively. These electrons and those furnished by the metal ions may have influenced the high electrical conductivity of the hybrid thin films.

Plate I: UV-Lamp fluorescence photo image of nanocomposite film

3.1 Electrical Conductivity

The results of the electrical conductivity **(**Table 2) show that the Kashikoko sample modified with phenylamine (0.3 g) had the highest electrical conductivity of 1818 S/cm and the Kashikoko sample modified with phenylamine 1g had the lowest electrical conductivity of 950 S/cm. EC was irrespective of the sample weight and thickness. All the samples show good electrical conductivity and can be used for conductive materials in electronics.

Table 2: Electrical Conductivity of Thin Film Nanocomposites

S/N	Samples (g)	Weight (g)	Electrical conductivity (S/cm) and Film/Water Ratio	(S/cm)
1	Kashikoko/PA/0.2	1.79	1667(1:30)	1664
2	Kashikoko/PA/0.3	0.74	1821(1:30)	1818
3	Kashikoko/PA/1.0	1.00	953(1:30)	950
4	Kaffin-Koro/PA/0.1	0.81	1532(1:40)	1529
5	Kaffin-Koro/PA/0.2	0.77	975(1:30)	972

3.2 Antimicrobial Analysis

After incubation for 24 h, the result was taken by measuring the diameter of the zone of inhibition for those biofilms that have activity **(Table 3)**. The antimicrobial analysis of the nanocomposite silver nitrate incorporated thin film was examined against selected bacterial pathogens (*E. coli, salmonella sp.,* and *S. aureus*) by measuring the zone of inhibition. In the world of antimicrobial substances/surfaces, the degree to which these materials are inhibitory can be of importance to the health of the consumer. The nanocomposite thin films show activities on different pathogenic

microorganisms probably, due to the diverse physical features observed. Results in this study revealed the Kashikoko sample, CMC/PA/1g/$AgNO_3$ respectively had 34 mm and 26 mm diameter of the zone of inhibition, corresponding to its inhibitory activity against *E. Coli*. However, there was no activity recorded against *Salmonella* while Kashikoko/CMC/EG/PA/0.2/$AgNO_3$, and Kashikoko/CMC/EG/PA/0.3/$AgNO_3$, respectively, had a diameter of the zone of inhibition which ranged from 26-32 mm. The highest inhibitory activity was recorded against *Salmonella*, which had 32 mm. The Kaffin-Koro/CMC/EG/PA/0.1g/$AgNO_3$ and Kaffin-Koro/CMC/EG/PA/0.2/$AgNO_3$ had diameter zone of inhibition in the range of 19-36 mm for *E. coli*, *Salmonella* and *S. aureus* with the highest activity recorded against *S. aureus*. Generally, the nanocomposite thin films show various degrees of antibacterial activity against the test organisms. The only exception was sample Kashikoko/CMC/EG/PA/1/$AgNO_3$, which had no activity against *Salmonella*. The film can be useful for active food packaging and biomedical applications such as wound dressings. Xinru *et al.* (2020) have reported CDP/CMC/TP composite thin films with antibacterial properties against *S. aureus, B. subtilis*, and *E. coli*. The prepared films were proposed for functional packaging applications. The inhibitory activities may be attributed to the synergistic effects of the silver ions and nanoclay adsorptive properties. Luis et al. (2019) reported a concentration-dependent inhibitory microbial activity of LDPE/Zeolite/$AgNO_3$ films. The high percentage of $AgNO_3$ gave good inhibitory activities while films without $AgNO_3$ show no activities. Research has shown that elemental silver is believed to inhibit the growth of microbes either as a release system for ions or as a contact active material (Shipra *et al.,* 2010). Nanocomposite films bearing silver compounds have been shown to exhibit high inhibitory activities against gram-positive and gram-negative bacteria (Shipra *et al.,* 2010). The report by Elele *et al*. (2020) and Azeh *et al.* (2021) on non-silver incorporated amino acid-modified nano clays had less inhibitory values against antibacterial.

Table 3: Diameter of the Zone of Inhibition (mm)

S/N	Sample	*E. coli*	*Salmonell*a	*S. aureus*
1	Kashikoko/PA/0.2/CMC/EG/$AgNO_3$	28.00	29.00	26.00
2	Kashikoko/PA/0.3/CMC/EG/$AgNO_3$	27.00	32.00	28.00
3	Kashikoko/PA/1/CMC/EG/$AgNO_3$	34.00	Nil	26.00
4	Kaffin-Koro/PA/0.1/CMC/EG/$AgNO_3$	19.00	28.00	36.00
5	Kaffin-Koro/PA/0.2/CMC/EG/$AgNO_3$	19.00	30.00	34.00

3.3 Meat Analysis

The total bacteria count of a substance is a quantitative estimate of the number of microorganisms present in a sample. This measurement is represented by the number of colony-forming bacteria units per gram (cfu/g) in the sample (Corrosionpedia (2019). Not all bacteria cells produced colonies as some bacteria tend to cluster and some are non-viable. For this reason, results are reported as colony-forming units (cfu/g) of bacteria culture. Ideally, only plates with 25-250 colonies are used. Counts above 250 are considered too numerous to count (TNTC) because it is impossible to tell whether colonies are separated whereas, plates with less than 25 colonies do not have a statistically significant number of colonies.

From the result above **(Table 4)**, Kashikoko/PA/0.3g/CMC/EG/$AgNO_3$ and Kaffin-Koro/PA/0.1g/ CMC/EG/$AgNO_3$, Kashikoko/PA/0.2g/CMC/EG/$AgNO_3$ and Kashikoko/PA/1g/CMC/EG/$AgNO_3$ were either extremely too numerous to count (ETNTC) or too numerous to count (TNTC). This implies that the thin film could not inhibit the growth of microbes on the meat.

The Kaffin-Koro//PA0.2g/CMC/EG/$AgNO_3$ sample had no total count, implying that the film inhibited the growth of microbes on cultured meat and could be useful for preserving meat.

Table 4: Meat Preservation Analysis using Thin Film Nanocomposites

S/No	Sample (g)	Total Count (Cfu/g)
1	Kashikoko/PA/0.2/CMC/EG/$AgNO_3$	LESS DENSE TNTC
2	Kashikoko/PA/0.3/CMC/EG/$AgNO_3$	TNTC
3	Kashikoko/PA/1/CMC/EG/$AgNO_3$	EXTREMELY TNTC
4	Kaffin-Koro/PA/0.1/CMC/EG/$AgNO_3$	TNTC
5	Kaffin-Koro/PA/0.2/CMC/EG/$AgNO_3$	NIL

3.4 FT-IR Spectra of Nanocomposite Film

The functional groups in the samples (S3 and S4) are depicted in Fig. 1 and Table 5. The absorption band between 3615 – 3250 cm^{-1} was due to the –OH stretching vibrations, which confirms the existence of a hydroxyl (OH) group in the sample named (S3) (Maheswari *et al.*, 2012; Siddiqui *et al.*, 2011).

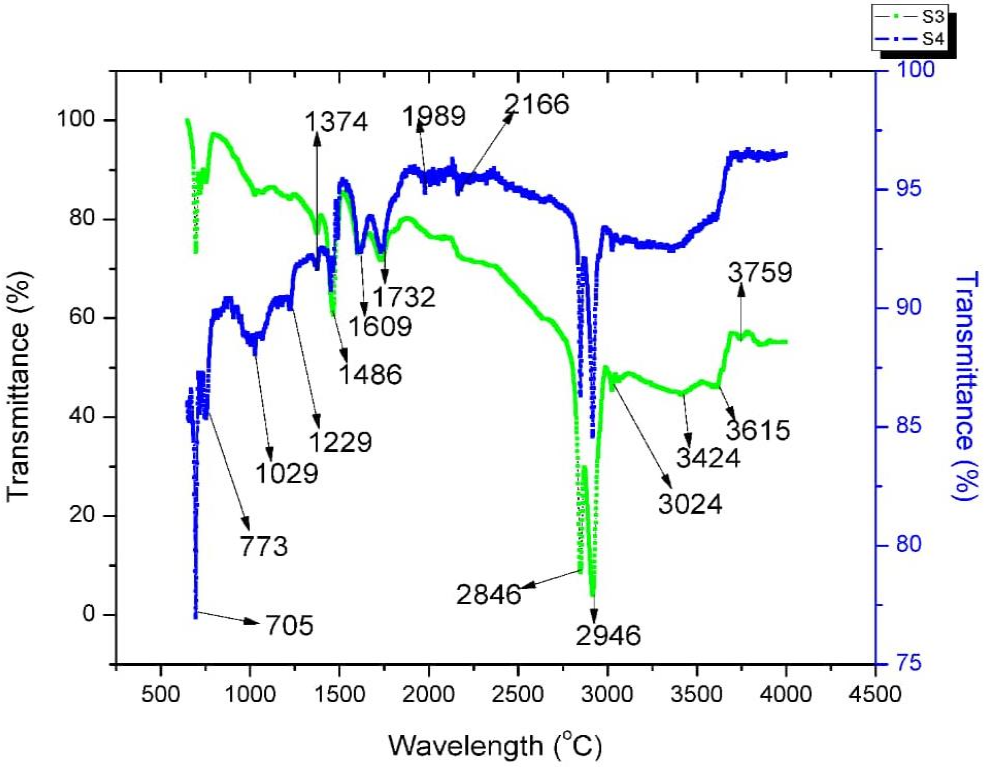

Fig. 1: Absorption for S3 and S4 Nanocomposite Thin Film

The band around 3424 - 3024 cm^{-1} is assigned to N-H stretching vibration (Pacheco *et al.*, 2012) and confirms the presence of amino acid in nano clay. The band around 2946 - 2846 cm^{-1}, shows the C-H asymmetric and symmetric stretch. The band corresponds to the C-H stretching vibration of the $–CH_2$ group in CMC and amino acids. The peak at 1470 cm^{-1} shows the bending vibration of C-H. The band at 720 cm^{-1} confirmed the presence of the C-H bond of the methylene group. The band at 1732 cm^{-1} is assigned to C=O due to the amide bond in organically modified nanoclay (Nondiyanto *et al.*, 2019). The free carboxylate group (COO-) was recorded at 1609 cm^{-1}. This also supports the presence of amino acids in clay. Peak at 1374 cm^{-1} and 1486 cm^{-1}, confirms the presence of C-H bend (methylene). The absorption band at 1229 cm^{-1} is due to aromatic CH_3 (plane bend) while the band at 1029 cm^{-1} has been assigned to Si-O-Si in clay. Whereas, absorptions at lower frequency regions indicate the presence of C-C, C-OH, C-O-C, and C-H (Phenyl) bonds, and those from the nanoclay (Coates, 2000; Azeh *et al.*, 2023).

This study shows that S3 and S4 samples can be used at high temperatures. DTGA (Differential thermal analysis) showed the inflection point, which revealed that samples S3 and S4 (Fig. 3) exhibited a single inflection temperature of 650 ^oC, which may be attributed to the nano clay layers in the CMC matrix. The DTG result shows that samples (S3 and S4) were stable up to 450 ^oC before sample mass began to lose at 650 ^oC. S3 chars at 750 ^oC while S4 chars at 800 ^oC implying that S3 was more stable than S4. This was similar to the findings by Azeh *et al.* (2023) on CMC/organoclay/PANI conducting nanocomposite thin film with antibacterial properties.

Table 5: FT-IR Assignment for Sample S3 and S4 Nanocomposite Thin Films

Sample	Wavenumber (cm^{-1})	Band Assignment	Reference
1 g Kashikoko modified with phenylalanine and silver nitrate			
	3615–3250	–OH bond stretching	Siddiqui et al. (2011); Maheswari et al. (2012)
	3424	$-NH_2$	Pacheco et al. (2012)
	3024	C–H Aromatic stretch	Nondiyanto et al. (2019)
	2946	C–H bond	Azeh et al. (2020)
	2846	C–H	Azeh et al. (2020)
	1486	C–H bond	Coates (2000)
0.1 g Kaffin/Koro modified with phenylalanine and silver nitrate			
	1732	C=O bond	Nondiyanto et al. (2019)
	1609	Conjugated C=C	Nondiyanto et al. (2019)

	1486	C–H bend	Coates (2000)
	1374	C–H rocking	Tufan et al. (2017)
	1229	CH_3	Nondiyanto et al. (2019)
	1029	Si–O–Si	Coates (2000)
	773	C–H	Azeh et al. (2023)
	705	C–C	Azeh et al. (2023)

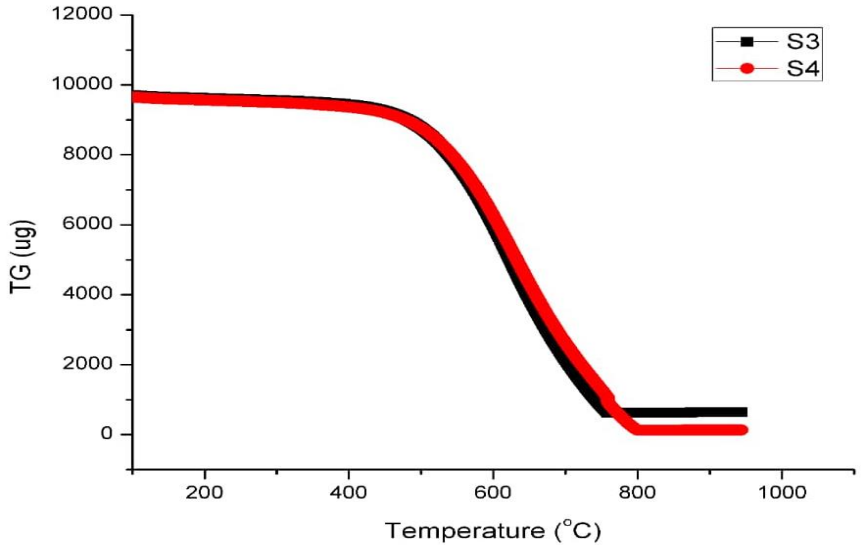

Fig. 2: TGA curve of S3 and S4 samples

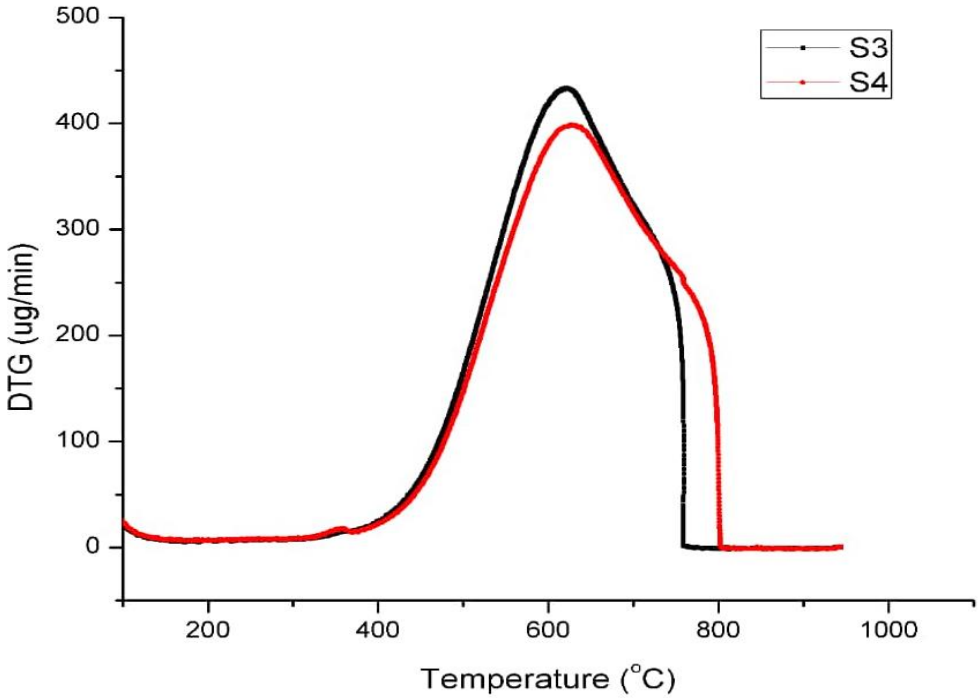

Fig. 3: DTG curve of S3 and S4 samples

3.6 Morphological Study of Nanocomposites Film using SEM

The scanning electron microscope (SEM) images of the S3 and S4 samples (Plate II) of the nanocomposite films were studied using SEM to confirm the structural changes that may occur due to nanoparticle present or differences in the structural arrangement due to the composition of the nanocomposite material and their interaction within the matrix bulk. The composite films showed good evidence of strong interactions between the organically modified nano clay particles and the matrix materials as shown by their smoothness, cellular web, lamellar and stacking morphology, and tridimensional network (Azeh *et al.*, 2020). The surface was rough due to nanoparticle agglomerations in the CMC matrix. Surfaces of this type are potentially viable for functional performance and may have contributed to the inhibitory properties of the films against bacteria. Despite the different nanoparticle compositions, the film displayed similarities in structure due to the presence of evenly distributed large and small pores. It was observed that the nanoparticle distribution in the matrix material appeared as white oval-shaped particles bound by the CMC matrix (Lexa, 2014).

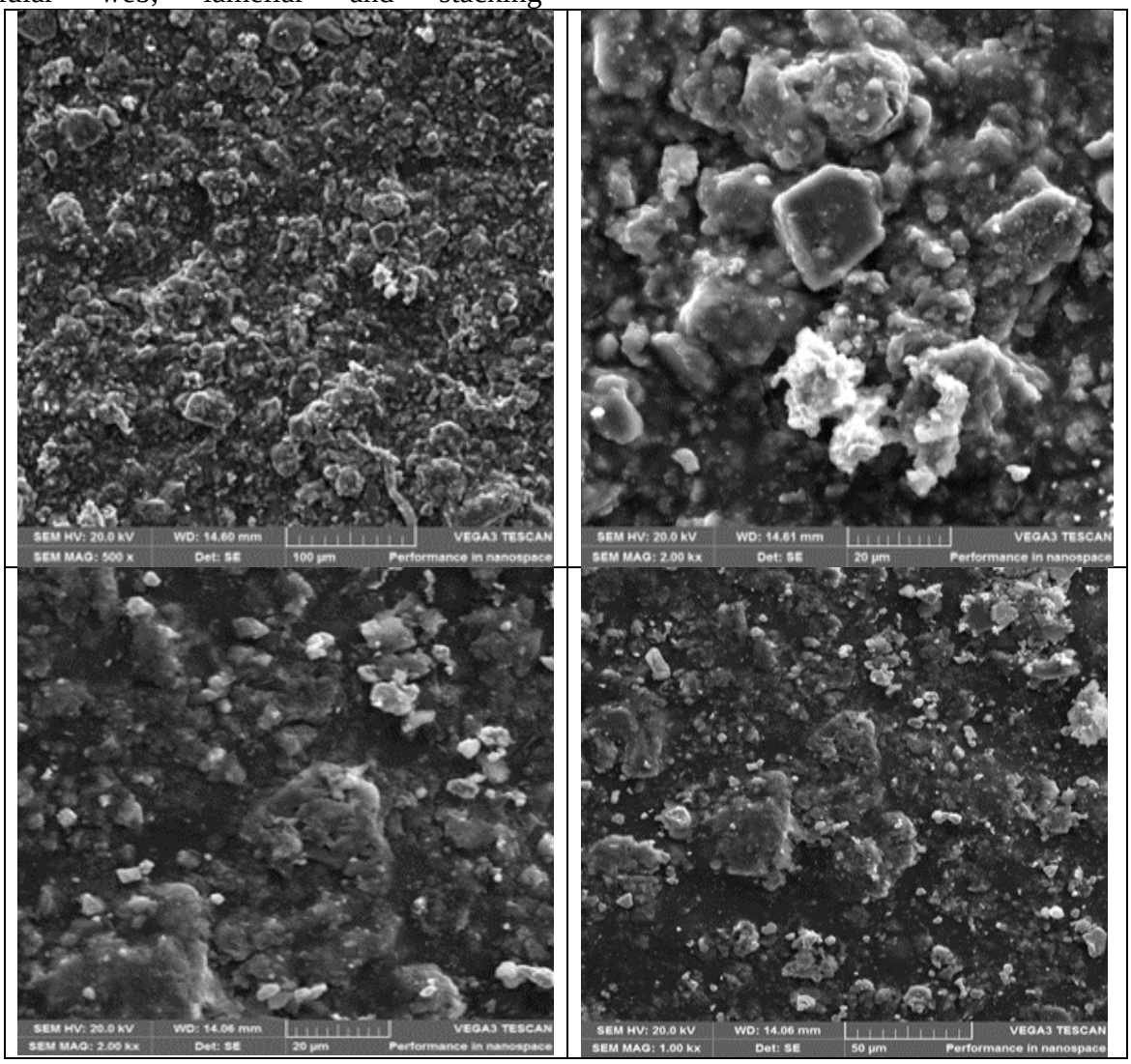

Plate II: SEM image of S3 and S4

4.0 Conclusion

The study successfully synthesized and characterized conductive antibacterial thin films using Kashikoko and Kaffin-Koro biopolymers, demonstrating promising results for their application in active food packaging and biomedical fields. The quantitative analysis revealed that silver nitrate significantly enhanced the films' electrical conductivity and antimicrobial properties. Specifically, the Kashikoko/PA/0.3 sample achieved an impressive conductivity of 1818 S/cm, while the largest inhibition zone of 34 mm against E. coli was observed in the Kashikoko/CMC/EG/PA/1g/$AgNO_3$ sample. These findings highlight the potential of biopolymer-based films as effective materials for preventing microbial contamination in various applications.

In conclusion, the synthesized conductive antibacterial films exhibit substantial electrical conductivity and antimicrobial activity against pathogenic bacteria. This dual functionality positions them as innovative solutions for enhancing food safety and providing protective measures in healthcare settings. The successful incorporation of biopolymers and silver nitrate into the films suggests that such materials could be further developed and optimized for commercial use.

It is recommended that future research focus on the scalability of the synthesis process to facilitate mass production. Additionally, long-term stability studies and comprehensive evaluations of the films' mechanical properties and biodegradability should be conducted. Investigating the films' performance in real-world conditions will also be essential for assessing their effectiveness and practicality in various applications.

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6.0 References

Abdul Rahman, A. M., Roshidi, M. H., & Shamsudin, N. (2023). Development of novel organoclay-based antimicrobial composites. *Journal of Polymer Science, 61*, 4, pp. 786-799.

Khan, M. A., Ali, M., & Kumar, A. (2023). The role of organoclay in enhancing the properties of polymer composites. *Composites Science and Technology, 215*, 109024.

Nithya, A., Manohar, S., & Varadharajan, M. (2023). Conductive polymer composites: Synthesis, properties, and applications. *Materials Today: Proceedings, 67*, 292-298.

Zhang, Y., Chen, J., & Wei, Q. (2023). Recent advances in antibacterial materials: Organoclay composites and their applications. *Journal of Hazardous Materials, 442*, 130083.

Asep, B., Nandiyanto, D., Rosi, O. & Risti, R. (2019). How to read and interpret FT-IR spectroscopy of organic material. *Indonesian Journal of Science & Technology*, 4(1), pp. 97-118.

Azeh, Y., Elele, U.U. & Alfred, G. (2021). Antibacterial evaluation of modified Nanoclay. *Int. J. Nanomater Mol Nanotechnol.*, 3(1), pp. 103-109.

Azeh, Y., Adetitun, D. O., Olatunji, G. A. & Adekola, F. A. (2020). Application of cellulose acetate-reinforced nanocomposite fluorescence film as filter and bio-packaging material with antibacterial properties. *Annals of Science and Technology - A*, 5(1), pp. 45-55.

Azeh, Y., Musah, M., John, T. M., Badeggi, U. M., Umar, G., Ahmed, T., Muhammad, Z. D., Oluwaseyi, D. S., Umar, M. T., Abdulkarim, M. A., Ibrahim, A. A., Muhammad, A. I. (2023b). In situ synthesis of polyaniline nanohybrid and formulation of polyaniline/carboxymethyl cellulose/ethylene glycol nanocomposite:

Study of its conducting and antibacterial properties. *J mate poly sci*, 3, 4, pp. 1-9.

Bahmid, N. A., Suloi, A. N. F., Engelen, A., Muslih, A. & Hernawan, A. (2024). Antimicrobial food packaging: Interaction of compounds and bacterial growth. *Curr Food Sci Tech Rep.*, 2, pp. 121–131: DOI: https://doi.org/10.1007/s43555-024-00024-w.

Coates, J. (2000). Interpretation of infrared spectra: A practical approach. *Encyclopedia of analytical chemistry*, 12, pp. 10815-10837.

Elele, U. U., Azeh, Y., Bala, E., & Alfred, G., (2020). Synthesis, characterization and in vitroantibacterial activities of some amino acids functionalized nanoclay from two local deposits. *International Journal of Research and Innovation in Applied Science, 5, 12,* pp. 147-158.

Fadiji, T., Rashvand, M., Daramola, M. O. & Iwarere, S. A. A. (2023). Review on antimicrobial Packaging for extending the shelf life of food. processes, 11, 590. doi: https:// doi.org/10.3390/pr11020590

Lexa, G. (2014). *Morphological, mechanical and rheological behavior of cellulose nanocrystalpoly (methyl methacrylate) nanocomposites prepared by wet ball milling and melt mixing.* M.Sc. Thesis Submitted to the School of Graduate Studies McMaster University.

Motelica, L., Ficai, D., Ficai, A., Oprea, O. C., Kaya, D. A., & Andronescu, E.(2020). Biodegradable Antimicrobial Food Packaging: Trends and Perspectives. Foods, 9(10), pp. 1438-1474. DOI:10.3390/foods9101438.

Maheswari, C.U., Reddy, K.O., Muzenda, E., Guduri, B. R. & Rajulu, A.V. (2012). Extraction and characterization of cellulose microfibrils from agricultural residue-*Cocos nucifera L*. *Biomass Bioenergy*, 46, pp. 555–563.

Pacheco, D. M, Johnson, J. R. & Koros, W. J. (2012). Aminosilane functionalized cellulosic polymers for increased carbon dioxide. *Industrial & Engineering Chemistry Research*,

Siddiqui, N., Mills, R. H., Gardner, D. J. & Bousfield, D. (2011). Production and characterization of cellulose nanofibers from wood pulp. *J. Adhes. Sci. Technol.*, 25(6–7), pp. 709–721.

Shaheen, M., Jain, A., Khan, W. & Domb, A. (2019). Antimicrobial material, an overview in antimicrobial materials for biomedical applications. Chapter 1. Antimicrobial materials: An Overview, pp. 1-37. doi: 10.1039/9781788012638-00001.

Shao, X., Sun, H., Jiang, R. & Yu, Y. (2020). Physical and antibacterial properties of corn distarch phosphate/carboxymethyl cellulose composite films containing tea polyphenol. *Journal of Food Processing and Preservation,* 44, 4, doi:10.1111/jfpp. -14401.

Tripathi, S., Mehrotra, G. K. & Dutta, P. K. (2011). Chitosan-silver oxide nanocomposite film: Preparation and antimicrobial activity. *Bulletin of Materials Science,* 34(1), pp. 29-35; doi:10.1007/s12 -034-011-0032-5.

Tufan, M, Caglagul, T. & Hasan, F.G. (2017). Preparation and characterization of carboxymethyl cellulose film from sunflower stalk, *International Journal of Advances in Science Engineering and Technology,* 5, 2, pp. 1-8.

Compliance with Ethical Standards

Declaration

Ethical Approval

Not Applicable

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Authors; Contribution

Azeh Yakubu, Musah Monday, Mathew John Tsado, and Ibrahim Fatima: Conceptualized, developed the method, executed the experimental, and wrote the first draft; Azeh Yakubu, Ameh Ohiga Alfa Ebune, Umar Musa Tanko, Badeggi Umar Muhammad, Abdulkarim Muhammed Awwal: Proofread the manuscript; Muhammad Aishetu Ibrahim, Ibrahim Ahmed Aliyu, Ismail Haruna[1], Muhammad Karima Tani and Nasiru Ibrahim: Purchased all the chemicals and reagents used for the experimental works.