iJRASET

International Journal For Research in
Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 9 Issue: VIII Month of publication: August 2021

DOI: <https://doi.org/10.22214/ijraset.2021.37430>

www.ijraset.com

Call: 08813907089

E-mail ID: ijraset@gmail.com

Preparation of Polypropylene/silver nanoparticles Nanocomposite films and Evaluation of its Barrier and Antimicrobial Properties for Packaging Applications

Rajesh Kumar Sahoo

Laboratory for Advanced Research in Polymeric Materials (LARPM), Central Institute of Plastics Engineering and Technology (CIPET), B-25, CNI Complex, Patia, Bhubaneswar, Odisha, India

Abstract: Polymeric films which can be used in packaging industries were prepared by blown film method with polypropylene chips and silver nanoparticles. The nanocomposite films were characterized concerning its potential use. Oxygen Transmission rate (OTR) measurements were done in order to ascertain permeability of oxygen through the polymeric films. It was found that the permeability of oxygen through nanocomposite film is higher than that of virgin polypropylene film due to incompatibility between polypropylene matrix and silver nanoparticles. The water vapour transmission rate (WVTR) test of the polymeric nanocomposite films was calculated in order to know the information about mass transfer mechanisms and solute-polymer interactions in the food packaging film. It has been found that, there is a significant decreased water vapour transmission rate through nanocomposite films compared to that of virgin polypropylene film. With increasing the concentration of silver nanoparticles, this effect was reduced. The explanation for this could be that with higher concentration of silver nanoparticles agglomeration in the polymer film could be the possibility which create narrow pathway for water molecules to travel. The effect of various silver nanoparticles content in the polymer nanocomposites with respect to its antimicrobial efficacy against the Gram positive bacteria *Escherichia coli* and Gram positive bacteria *Staphylococcus aureus* were studied. However, *Escherichia coli* was most effectively retarded by silver nanoparticles than *Staphylococcus aureus*, which is related to the difference in the cell wall of the gram positive and gram negative bacteria.

Keywords: Polypropylene, Silver nanoparticles, permeability, *Escherichia coli*, *Staphylococcus aureus*, micro-bacterial activity

I. INTRODUCTION

Polymers have become necessary in modern life and the material of choice in packaging applications. It is because of their high performance, good processability, and low cost, light weight. It is projected that by 2050 the plastic production will exceed 300 million metric tons. Durability of plastic employed in packaging applications is of utmost important for its long lasting use and disposability with respect to environmental concerns. As plastic films used for less than a week or month, the durability is the utmost resources for minimizing growth of disposed plastics as pollution waste. By preparing plastics of high barrier performance and if possible to reuse and recycle we can minimize the deposition of waste plastic materials in oceans and landfills [1]. The main requirement for plastic materials to be used in various packaging purposes are of good barrier performance to atmospheric pollutants and environmental gases like oxygen, carbon dioxide and water vapour along with enhanced mechanical properties and transparency. In recent times the most used plastic materials in packaging industry are polypropylene (PP), poly (ethylene terephthalate) (PET) and polyethylene (PE). It is reported in many review research papers that by mixing nanofillers has improved the barrier properties of nanocomposite films [1-2]. Recent progresses in polymer nanocomposites have attracted attention because of the promises offered by this technology to enhance the barrier properties of low-cost commodity polymers. Many studies have demonstrated improvements in permeability reduction to gases, moisture and organic vapours resulting from the addition of low concentration metal nanoparticles to different polymeric matrixes. Nanoparticles having low volume fraction influences barrier properties of the polymers. This is due to nano meter size and intra-particle distances. The desired permeability values of the polymer nanocomposites are usually reached at low filler volume fraction, allowing microscopic dispersion and low density of the polymer. Yano et al. obtained a very good reduction in water vapour permeability coefficient of a polyamide containing 2 weight % of mica as filler in comparison to virgin polymer [3].

Similarly Avella et al. suggested that the calcium carbonate nanofillers significantly reduces the permeability of oxygen and carbon dioxide for PP nanocomposites [4]. The improvement in barrier properties of the nanocomposite by addition of nanoparticles is possibly due to the tortuous path model.

The mechanism says that the presence of nanoparticles generates an overlapped structure that hinders penetrant diffusion and thus decreases the permeability of the materials [4-6]. In recent times use of nanotechnology in preparing polymer nanocomposite due to their inherent properties is comprehensively and creatively used in many fields, particularly in medical devices, health care products. Infections are produced when people touch, eat or drink something that is contaminated with germs. Every day we use many materials including food packaging items for our daily need. These packaging items are infected by many harmful bacteria; antimicrobial packaging is thought to be a subset of active packaging [5-6].

It is the promising technology which effectively impregnates the antimicrobial agent into the food packaging materials which subsequently delivers over the stipulated period of time to kill the pathogenic microorganisms affecting food products thereby increasing the shelf life of food materials [7].

Therefore incorporation of antimicrobial agents into food packaging materials has received considerable attention. An antimicrobial nanocomposite film is particularly attractive due to its suitable structural integrity. Materials in the range of nanoscale have higher surface to volume ratio as compared to their micro scale counterparts. This allows nanoparticles to attach more in numbers to the microorganisms, which confers greater efficiency [8-9]. The antimicrobial film used for food packaging applications are mainly based on silver (Ag), which is well known for its strong toxicity to a wide range of pathogenic bacteria and fungi. Recently, silver nanoparticles (Ag-NPs) based antimicrobial polymeric materials have attracted considerable research interest [9-11]. Several research papers have reported that the antibacterial effect of Ag-NPs is largely related to its particle size (usually <100 nm), shape and particle dispersion within the polymer [11-15].

Metallic Ag is considered to be a non-reactive material but in this physical state it can chemically combine with moisture and getting ionized to form highly reactive silver ions (Ag^+) [11,15]. The Ag^+ ions can attach to negatively charged components in proteins and nucleic acids, causing structural variations in the cell membranes. Many antimicrobial polymer nanocomposite films have been prepared by melt mixing method [15-17].

Literature review reveals the preparation of nanocomposite films incorporated with Ag-NPs with various polymers like polypropylene (PP) [17-18], polyurethane [19-20], polyester [21-22], polyamide [23-24] and polyacrylate [25] by melt compounding technique. This melt compounding method is the most efficient method for the preparation of nanocomposite film for antimicrobial applications, compared to other methods like conventional deposition of metallic particles directly on the surface of the substrate, solution blending and vapour coating.

Polypropylene-silver nanoparticles (PP/Ag-NPs) nanocomposite film with low release potential of Ag^+ ion shows outstanding long term antimicrobial activity. It has been confirmed that the prolonged and steady release of Ag^+ ions in the aqueous atmosphere is the reason behind its antimicrobial activity. Ag-NPs can progress the water penetration characteristics of the nanocomposite film either by generating some additional voids within the nanocomposite film or dropping the crystallinity of the same to permit the access of more water molecules [26].

In the present investigation, PP/Ag-NPs nanocomposite films were prepared at the different weight percentage of Ag-NPs as filler and polypropylene grafted maleic anhydride (PP-g-MAH) as compatibilizer by the melt blending method. The potential use of the prepared nanocomposite film for food packaging applications were evaluated by investigating the gas barrier and antimicrobial properties.

II. MATERIALS

The polypropylene (PP) homopolymer pellets (Repol H100EY) of density 0.96 and Melt Flow Index 11g/10min (230°C/2.16 kg, ASTM D 1238) supplied by Reliance Polymers was used in the preparation of composite film which meet the FDA requirements for all food contact and cooking application in the Code of Federal Regulations in 21 CFR 177.1520. Maleic anhydride grafted polypropylene has been used as a compatibilizer to help a good bonding between silver nanoparticles (Ag-NPs) and polymer matrix. The commercial grade Ag-NPs used in this experimental process is of 99.9% purity. It has average particle size ranging from 50nm to 80 nm with specific surface area 5.37m²/gm. The Ag-NPs were fine powder with bulk density of 0.312 gm/cm³ and true density of 10.5 g/cm³. The morphology of the nanoparticles is spherical of one with cubic crystallographic morphological structure. It has been purchased from Nanoshel, Intelligent Materials Pvt. Ltd., Panchkula, Haryana, India.

III. PREPARATION OF POLYPROPYLENE AND SILVER NANOCOMPOSITE FILMS

Prior to extrusion, the Ag-NPs powder was dried in a vacuum chamber oven at 80°C for 12 hours to remove the absorbed moisture. Three PP master batches were prepared containing PP and Ag-NPs along with virgin PP master batch. The composition of each master batch containing Ag-NPs was in the range of 0.5%, 1%, and 2% by weight without using compatibilizer. Table-1 represents the master batch composition of different samples. Nanocomposite films were prepared by melt mixing of the two components using Torque rheometer (Haake Rheomix OS, Germany) with counter rotating roller rotors having a chamber size of 66 cm³. The screw speed was 100 rpm and the mixing time was 15 minutes for all the compositions. The barrel temperature profile was optimized from 175°C to 190°C from feed to die zone. After compounding, the mixed material was extruded with the help of blown film extrusion set up to produce cylindrical films. The mechanical test was performed to select the optimum filler loading. The optimized composition of the master batch was further mixed with different wt. % of PP-g-MAH as compatibilizer. The mixture was extruded after passing through twin screw extruder to homogenize the mixture thoroughly to get the pellets. The prepared pellets were further processed to obtain the desired film samples with the help of blown film extrusion set up for property evaluation.

Table-1: Composition of developed PP/Ag-NPs nanocomposite films

Sample	Weight %		
	PP	PP-g-MAH	Ag-NPs
Virgin PP	100	0	0
PP/Ag-NPs	99.5	0	0.5
PP/Ag-NPs	99	0	1
PP/Ag-NPs	98	0	2
PP/ PP-g-MAH /Ag-NPs	94	5	1

IV. CHARACTERIZATION

A. Oxygen Transmission rate (OTR) Measurement

Virgin PP and PP/PP-g-MAH/Ag-NPs nanocomposite film systems with different Ag-NPs concentration were performed with an Oxygen Permeation Analyzer (Illinois Instruments, Model 8501) according to ASTM D-3985. A blown film of uniform thickness for each specimen (less than 2 µm variation) was mounted as a sealed semi barrier between two chambers at ambient atmospheric pressure, 23°C in dry environment. Nitrogen gas containing 2 % hydrogen was slowly purged in one chamber and another chamber contained oxygen gas. As oxygen gas permeated through the film into the nitrogen carrier gas, it was detected by coulometric sensor. Oxygen transmission rate (OTR) was calculated considering the following equation:

$$OTR = \frac{E_e - E_o(Q)}{A \times R_L} \quad (1)$$

Where E_e is steady-state voltage level, E_o is zero voltage level, A is specimen area, Q is calibration constant and R_L is value of load resistance. Measurements were performed at controlled temperature and repeated twice. The average values of permeability were estimated.

B. Water Vapour Transmission Rate (WVTR) Measurement

The permeability of the sample was measured using the ASTM E-96 method in PBI-Dansensor L 80-5000. All samples were cut from film circularly and mounted for evaluation of WVTR for each sample. Two replicates for each sample were measured. A dry room with a specified relative humidity is separated by a wet room, where the atmosphere is saturated with water vapour at a known temperature, through a sheet of material to be tested. Changing humidity in the dry chamber is achieved by water vapour passing through the tested material and it is detected by a humidity sensor that is able to provide an electrical signal that is measuring relative humidity in the dry room. The time required to increase humidity to a certain amount is measured and converted into a transmission rate of water vapour. The permeability of the samples was measured at 23±2°C.

C. Measurement of Antibacterial Properties by Plate Count agar (PCA) Technique

Plate count agar (PCA) technique also called standard Methods Agar (SMA) is a microbiological growth medium commonly used to assess or to monitor total viable bacterial growth of a sample. The test followed the ASTM E2149 standard method. Normally microbial growth medium contains 0.5 % peptone, 0.35 % yeast extract, 0.1 % glucose, 1.5 % agar. P^H was maintained as neutral during the experimental process. Two typical food pathogens including one Gram-positive bacteria, *Staphylococcus aureus* ATCC-14458, and one Gram-negative bacteria, *Escherichia coli* ATCC-11775 were separately inoculated on PP-Ag-NPs samples with different wt % of Ag-NPs loading. A 10 fold serial dilution technique was used for ensuring a reliable accounting of the bacteria colonies (usually ranging from 30 to 300 colonies). Hundred micro litres of bacterial suspension after the shaking were placed over the agar into sterilized Petri dishes. This method is used for evaluating the influence of the presence of NPs on polymer matrix on the antimicrobial properties. The inoculated plates were then cultivated at $37^{\circ}C \pm 0.5^{\circ}C$ for 24 hour before calculating the viable cell count of the testing bacteria and evaluating the antibacterial efficacies using equation shown below

$$\% R = (A-B)/A \times 100 \quad (1)$$

Where R is the reduction of bacteria (%), A is average number of bacterial colonies from PP matrix without silver nanoparticles (CFU/ml); B is average number of bacterial colonies from PP matrix incorporated with silver nanoparticles (CFU/ml).

V. RESULTS AND DISCUSSION

A. Oxygen Permeability Measurement

The results of measurements of oxygen permeability for the virgin PP and PP/PP-g-MAH/Ag-NPs (94:5:1) nanocomposite films are collected in table-1 and represented in figure-1. The oxygen transmission rate (OTR) of the nanocomposite film shows an increase with respect to virgin PP film. It is observed that the increase in OTR is more significant with the increase in Ag-NPs loading. This is due to the lack of interaction between the PP chains and the Ag-NPs that might lead to the formation of even more pronounced voids in the structure, which would boost the gas diffusivity. The addition of compatibilizer (5 wt. %) to 1 wt. % Ag-NPs loaded polypropylene matrix has shown decrease the OTR value from $3633.7 \text{ cm}^3(\text{STP})/\text{cm}^2\text{day}$ to $3595.2 \text{ cm}^3(\text{STP})/\text{cm}^2\text{day}$ in comparison to PP/1wt.% Ag-NPs nanocomposite film (8.47 % reduction). It indicates that compatibilizer has slightly improved the mixing of Ag-NPs within the polymer matrix. In general OTR of the PP/PP-g-MAH/Ag-NPs (94:5:1) nanocomposite film shows an increased permeability with respect to the virgin PP film. The percentage increase is 4.28% for 0.5% loaded Ag-NPs nanocomposites, 9.66 % for 1.0% loaded Ag-NPs nanocomposites and 12.33 % for 2.0% loaded Ag-NPs nanocomposites. This is due to improper dispersion of Ag-NPs in the polypropylene matrix and existence of some metal agglomerates. These agglomerates might have show the way for the formation of micro porosity in the polymer film, which would enhance the diffusion of gas molecules [1-2] . Besides these, other factors which also play vital role on the transport properties of gases are filler type, aspect ratio, the filler-induced crystallinity, the polymer chain immobilization, the filler-induced solvent retention and porosity. So, the presence of Ag-NPs did not significantly modify the oxygen transmission rate of nanocomposite films. Compton et al. has reported that a decrease of permeability higher than the theoretical decrease during their study in in-situ preparation of palladium and palladium-silver alloy nanoparticles in a polyimide matrix [27] . They accredited this reduction in permeability by the presence of crystalline nanoparticles and by the formation of strong interface between polymer matrix and nanoparticles. Further, the decrease of barrier properties can be explained by a plasticization effect of the polymer matrix due to the increase of water molecules sorbed by the polymer as the water activity increased. This helped to minimize the contribution of the strong interface between the polymer matrix and nanoparticles presented at lower water activity.

Table-2: Oxygen permeation value of virgin PP and its nanocomposite film samples at different Ag-NPs loading

Sample	O ₂ permeability (cm ³ (STP)cm ² day)	Percentage of increase of oxygen permeation through nanocomposite in comparison to virgin PP
Virgin PP	3315.27 ±165.76	
PP/0.5wt.% Ag-NPs	3457.35±172.86	4.28 %
PP/1wt. % Ag-NPs	3633.7±181.68	9.6 %
PP/2 wt.% Ag-NPs	3723.83±186.19	12..33%
PP/PP-g-MAH/Ag-NPs(94:5:1)	3595.92±179.79	8.47%

Figure-1: Oxygen permeability data of the virgin PP and PP/Ag-NPs nanocomposite films

B. Water Vapour Permeability of the Films

The data relating to WVTR of the virgin PP and nanocomposite films are summarized in table-2. It reveals that the nanocomposite films have shown reduction in WVTR by ~27 to 57% for different concentration of Ag-NPs loading with respect to the virgin PP film. But with the increased Ag-NPs loading i.e. of 0.5% to 1 % and then to 2% in polymer matrix has shown increased WVTR monotonically by 13.33% and 16.34%. But compatibilized nanocomposite has shown improved WVTR (8.31%) as compared to 2 wt. % Ag-NPs loaded PP matrix. This might be due to the reason that, with the increase in filler loading there is formation of voids near the filler-matrix interface and thereby decreasing the dispersion level of nanoparticles in the matrix. Similar to these results reported that higher Ag-NPs enhanced the moisture content of polymer films [30-32]. Similar reports were found in other films, such as agar/lignin/Ag-NPs and gelatine/Ag-NPs nanocomposite films [33-34]. WVTR of starch- CuO and Starch-Ag-NPs films was gradually decreased with increasing the weight percentage of CuO and Ag-NPs as reported by [35] but Qin and Liu et al. reported a contrasting result for chitosan/Ag-NPs nanocomposite films [36]. From the table-2, it is revealed that water vapour absorption rate was reduced on addition of compatibilizer because of its participation in improving the degree of dispersion of Ag-NPs in the polymer matrix. It is realistic that the water permeability depends on the structure of Ag-NPs and its random arrangements inside the nanocomposite film [33]. The uniform dispersed structure with less aggregation of filler particles can absorb less water, which results in improvement of barrier property. Since one of the most significant purpose of a film for use in food packaging application is to decrease the transfer of moisture from the surrounding environment in to the food particles. Therefore estimation of the film permeability to water vapour is crucial, and this property must remain at lowest feasible amount [38].

Table-3: Water vapour transmission rate of virgin PP and its nanocomposite film samples at different Ag-NPs loading

Sample	WVTR (gm cm/m ² day)	Percentage of Reduction of WVTR of nanocomposite in comparison to virgin PP
Virgin PP	0.30833±0.015	
PP/0.5wt.% Ag-NPs	0.13484±0.006	56.16 %
PP/1wt. % Ag-NPs	0.15286±0.007	50.32%
PP/2 wt.% Ag-NPs	0.17799±0.008	42.20%
PP/PP-g-MAH/Ag-NPs(94:5:1)	0.16409±0.018	46.75%

Figure - 2: Water vapour transmission rates of the virgin PP and PP/Ag-NPs nanocomposite films

C. Antimicrobial Properties

Quantitative assessment has been made to measure the effects of Ag-NPs in PP nanocomposite films by a plate-count-agar (PCA) technique using colonies of Gram-negative bacteria *Escherichia coli* ATCC 6538 and Gram-positive bacteria *Staphylococcus aureus* ATCC 8379. These two bacteria were separately inoculated on PP/Ag-NPs samples with different composition formulations. Count of the colonies was performed for control times of 0 hour and 24 hours for both virgin polymer and nanocomposite films. The overall antimicrobial activity of different PP nanocomposites containing silver nanoparticles improves in a significant way [39]. The comparative analysis of the antimicrobial activity of PP/Ag-NPs with different concentration of Ag-NPs and compatibilizer against gram positive bacteria *Staphylococcus aureus* and gram negative bacteria *Escherichia coli* is epitomised in table-5 and its graphical representation has been represented in figure-3. From data analysis it is seen that, antimicrobial activity against *Staphylococcus aureus*, the virgin polypropylene shows a control count of 201350 CFU ml⁻¹ colony forming units per ml [log CFU ml⁻¹ = 5.303] and this value is reduced up to values of 24000 [log CFU ml⁻¹ = 4.38] after 24 hours. The antimicrobial test of PP/Ag-NPs (0.5 wt. % of Ag-NPs) gives a bacteria colony count of 650 log CFU ml⁻¹ [log CFU ml⁻¹ = 2.81] after 24 hour. This shows an antibacterial activity of 1.57. This value represents a significant antibacterial activity against *Staphylococcus aureus*.

Table - 4 : Antimicrobial activity values (colony forming units per ml , CFU ml⁻¹ and log [CFUml⁻¹] of the inoculation and virgin PP sample at a control time of 0 and 24 hour against *Staphylococcus aureus* and *Escherichia coli*

		Staphylococcus aureus	Escherichia coli
		CFU ml ⁻¹	CFU ml ⁻¹
Inoculation	CFU ml ⁻¹	253,000	340,000
	log [CFUml ⁻¹]	5.403	5.531
Time = 0 hour	CFU ml ⁻¹	201,350	185500
	log [CFUml ⁻¹]	5.303	5.286
Time = 24 hour	CFU ml ⁻¹	24000	28500
	log [CFUml ⁻¹]	4.380	4.454

As per the PP/Ag-NPs nanocomposites is concerned with varying concentration of Ag-NPs, it is seen that a significant improvement in antimicrobial activity against the gram positive bacteria *Staphylococcus aureus* with antimicrobial activity value ranging from 2.81 to 2.47 for filler loading from 0.5 wt.% , 1 wt.% , 2 wt.% and 1 wt.% with compatibilizer respectively. The antimicrobial properties of the same sample against the gram negative bacteria *Escherichia coli* is also remarkable with antibacterial activity ranging from 2.93 to 3.53. The mechanism behind the possible antimicrobial activity shown by Ag-NPs is as follows:

- 1) Destruction of lipopolysaccharide cell wall leading to increased cell permeability, and considerable add-on to the respiratory enzyme present in the lipopolysaccharide cell membrane. It also attached to the receptor of the same. As we know cell membrane of most of the bacteria contains polymers with negatively charged chemical substance or group, there is an every possibility to that they can absorb metallic cations (Ag⁺ ion). Hence it is postulated that the cell membrane is the site at which metals exert bacterial toxicological activity [39-41].
- 2) Destruction to DNA leads to interruption in replication of the microorganism, and hinders in the work of ribosome and helps in preventing production of ATP.

Further, it is also ascertained that gram positive bacteria (*Staphylococcus aureus*) have thicker peptidoglycan layer that could prevent the microorganism to enter into the cytoplasm in more systematic way than that of gram negative bacteria (*Escherichia coli*). This has also been reflected from the comparison data of antibacterial activity value of nanocomposites(see Table-5).

Table - 5: Antimicrobial activity values (colony forming units per ml , CFU ml⁻¹ and log [CFUml⁻¹] of the inoculation and PP nanocomposites at different weight percentage of Ag-NPs at a control time of 0 and 24 hour against Staphylococcus aureus and Escherichia coli

Sample	Staphylococcus aureus		Escherichia coli	
	CFU ml ⁻¹	log [CFUml ⁻¹]	CFU ml ⁻¹	log [CFUml ⁻¹]
PP/0.5wt.% Ag-NPs	650	2.81	870	2.93
PP/1wt. % Ag-NPs	475	2.67	980	2.99
PP/2 wt.% Ag-NPs	450	2.65	3400	3.53
PP/PP-g-MAH/Ag-NPs(94:5:1)	300	2.47	900	2.95

Figure-3 : Comparative analysis of the antimicrobial activity of PP/Ag-NPs with different concentration of Ag-NPs and compatibilizer against gram positive bacteria Staphylococcus aureus and gram negative bacteria Escherichia coli

Regarding, PP/Ag-NPs samples with silver nanoparticles, it is important to remark a significant antimicrobial activity against Staphylococcus aureus with antimicrobial activity values of 1.5 and 1.7 for PP/Ag-NPs (1 wt. % and 2 wt. %) respectively. The antimicrobial activity of these nanocomposites against Escherichia coli is also significant with antimicrobial activity values close to 1.5 for both nanocomposite samples. Figure-4 and 5 represents the growth of Staphylococcus aureus after an incubation period for 24 h in a 37°C environment for virgin PP film and PP/Ag-NPs nanocomposite films in order of increasing concentrations of Ag-NPs at a contact time of 90 minutes respectively.

(Virgin PP film)

Figure -4: Represents the growth of bacteria Staphylococcus aureus after incubation for 24 h in a 37°C environment for virgin PP film

(PP/Ag-NPs nanocomposite film)

Figure-5: Represents the growth of bacteria *Staphylococcus aureus* after incubation for 24 h in a 37°C environment for PP/Ag-NPs nanocomposite film in order of increasing weight % Ag-NPs at the contact time of 90 minutes.

Figure-6 represents the growth of *Escherichia coli* after an incubation period for 24 h in a 37°C environment for virgin PP film. Figure- 7, 8 and 9 represent the growth of *Escherichia coli* after an incubation period for 24 h in a 37°C environment PP/Ag-NPs nanocomposite films in order of increasing concentrations of Ag-NPs at a contact time of 90 minutes. From the photographs stable microbial growths was seen because there was no delimiting and swelling appeared on the Muller Hinton agar (MHA) plate broth in the presence of *Escherichia coli* and *Staphylococcus aureus* for different Ag-NPs loaded nanocomposite films. The MHA plates were inoculated by swabbing these bacterial pathogens to create a confluent lawn of bacterial growth. The observations indicated that intracellular components of the strain reduced Ag⁺ ions as observed by change in colour of the sample from pale yellow to brown within 24 hours of incubation. It is further noted that the intensity of the colour increased up to 24 hours but maintained throughout the 72 hours period of observation. This indicates the killing of bacteria and antimicrobial action of silver ions [32].

(Virgin PP film)

Figure-6: Represents the growth of bacteria *Escherichia coli* after incubation for 24 h in a 37°C environment for virgin PP

(PP/Ag-NPs nanocomposite film)

Figure 7: Represents the growth of bacteria *Escherichia coli* in PP/(0.5 wt. %) Ag-NPs nanocomposites at the contact time of 90 minutes in a 37°C environment

(PP/Ag-NPs nanocomposite film)

Figure- 8: Growth of bacteria *Escherichia coli* in PP/ (1 wt. % Ag-NPs nanocomposites at the contact time of 90 minutes in a 37°C environment

(PP/Ag-NPs nanocomposite film)

Figure- 9: Growth of bacteria *Escherichia coli* in PP/ (2 wt. %) Ag-NPs nanocomposites at the contact time of 90 minutes in a 37°C environment

VI. CONCLUSION

Virgin PP film and different wt. % of Ag-NPs loaded nanocomposite films have been prepared using the blown film method and their barrier and antimicrobial properties have been investigated. Oxygen permeability measurement revealed that the nanocomposite film showed ~5% increase in OTR as compared to virgin PP. WVTR of virgin PP was decreased by 50 % on addition of 1 wt. % Ag-NPs. This showed the significant improvement in WVTR of the nanocomposite film. The PP/Ag-NPs nanocomposite films showed outstanding antimicrobial efficacy as documented by the percentage of the viable count reduction of the growth of *Escherichia coli* and *Staphylococcus aureus*. The virgin PP film did not inhibit the bacterial growth. However, the degree of antibacterial activity of the nanocomposite films was dependent on the percentage of Ag-NPs loading within the polymer matrix. Among all the films, the PP/PP-g-MAH/Ag-NPs (94:5:1) nanocomposite film showed a strong bactericidal effect against both *Escherichia coli* and *Staphylococcus aureus*. This is because of accumulation of Ag-NPs accumulate in the bacterial cytoplasmic membrane causing a significant increase in membrane permeability and leading to cell death. Hence, PP/Ag-NPs nanocomposite film with strong antimicrobial activity against both Gram-positive and Gram-negative bacteria may have good potential for using as antimicrobial food packaging materials. In conclusion, the lowest moisture content, oxygen permeability and antimicrobial properties of the films is a favourable assets regarding their application for the food packaging industry. Because these type of films can resist high level of atmospheric moisture and gaseous particles during handling, distribution, processing and storage of food products.

VII. ACKNOWLEDGEMENT

The study was carried out without any funding from any Institution/Organisation neither from Government or Private. I would like to thank all the staff of Laboratory of Advance Research in Polymeric Materials (LARPM), CIPET, Bhubaneswar, Odisha for providing necessary experimental support and also thank to Odisha University of Agricultural Technology (OUAT), Bhubaneswar for extending help for the antimicrobial tests and technical support.

REFERENCES

- [1] S. Lange, T. Arroval, R. Saar, I. Kink, J. Aarik and A. Krumme, "Oxygen barrier properties of Al₂O₃- and TiO₂-coated LDPE films. Polymer-Plastics Technology and Engineering," vol. 54, pp. 301-304, Feb. 2015, <https://doi.org/10.1080/03602559.2014.977426>
- [1] J. Lange and Y. Wyser, "Recent innovations in barrier technologies for plastic packaging—a review. Packaging Technology and Science: An International Journal," vol. 16, pp. 149-58, Jul.2003, <https://doi.org/10.1002/pts.621>.
- [2] K. Yano, A. Usuki, A. Okada, T. Kurauchi and O. Kamigaito, "Synthesis and properties of polyimide-clay hybrid. Journal of Polymer Science Part A: Polymer Chemistry," vol. 31, pp.2493-2498, Sep.1993, <https://doi.org/10.1002/pola.1993.080311009>
- [3] M. Avella, S. Cosco, M.L. Di Lorenzo, E. Di Pace, M.E. Errico and G. Gentile, "Nucleation activity of nanosized CaCO₃ on crystallization of isotactic polypropylene, in dependence on crystal modification, particle shape, and coating. European Polymer Journal," vol.42, pp. 1548-1557, Jul.2006, <https://doi.org/10.1016/j.eurpolymj.2006.01.009>.
- [4] Z. Duan and N.L. Thomas, "Water vapour permeability of poly (lactic acid): Crystallinity and the tortuous path model. Journal of Applied Physics," vol.115, pp.064903, Feb.2014, <https://doi.org/10.1063/1.4865168>.
- [5] G. Choudalakis and A.D. Gotsis, "Permeability of polymer/clay nanocomposites: a review. European polymer journal," vol.45, pp. 967-984, Apr.2009, <https://doi.org/10.1016/j.eurpolymj.2009.01.027>.
- [6] S. McKay, P. Sawant, J. Fehlberg and E.Almenar "Antimicrobial activity of orange juice processing waste in powder form and its suitability to produce antimicrobial packaging. Waste Management," vol.1, pp. 230-239, Feb.2021, <https://doi.org/10.1016/j.wasman.2020.11.048>.
- [7] D. Merchan, M. Agila, M. Arteaga and M. Criollo, "Antimicrobial Packaging: Food safety and product quality. International Journal of Research in Business and Social Science," vol.15, pp. 479-484, Jun. 2021, <https://doi.org/10.20525/ijrbs.v10i4.1251>.
- [8] T.Ratvijitvech and S.N. Pombejra, "Antibacterial Efficiency of Microporous Hypercross linked Polymer Conjugated with Biosynthesized Silver Nanoparticles from Aspergillusniger. Materials Today Communications," vol. 2, pp. 102617, Jul. 2021, <https://doi.org/10.1016/j.mtcomm.2021.102617>.
- [9] L.M. Stabryla, K.A. Johnston, N.A. Diemler, V.S. Cooper, J.E. Millstone, S.J. Haig SJ and L.M. Gilbertson, " Role of bacterial motility in differential resistance mechanisms of silver nanoparticles and silver ions. Nature Nanotechnology," pp.1-8, Jun. 2021.
- [10] C.Yang, R. Jian, K. Huang, Q. Wan and B. Feng B, "Antibacterial mechanism for inactivation of E. Coli by AgNPs @ polydoamine/titania nanotubes via speciation analysis of silver ions and silver nanoparticles by cation exchange reaction, Microchemical Journal," vol.1, pp.105636, Jan.2021, <https://doi.org/10.1016/j.microc.2020.105636>.
- [11] J.K. Sohal, A. Saraf and K.K. Shukla, "Silver Nanoparticles (Ag-NPs): Methods of Synthesis, Mechanism of Antimicrobial Action and Applications. Multidisciplinary Research and Development," vol.57, 2021:57.
- [12] K. Prusty and S.K.Swain, "Polypropylene oxide/polyethylene oxide-cellulose hybrid nanocomposite hydrogels as drug delivery vehicle. Journal of Applied Polymer Science," vol. 138, pp. 49921, Mar. 2021, <https://doi.org/10.1002/app.49921>.
- [13] Z. Ghorbani, N. Zamindar, S. Baghersad, S. Paidari, S.M. Jafari and L. Khazdooz, "Evaluation of quality attributes of grated carrot packaged within polypropylene-clay nanocomposites. Journal of Food Measurement and Characterization," vol.23, pp. 1-1, Apr.2021, <https://doi.org/10.1007/s11694-021-00925-7>.
- [14] J.M. Mata-Padilla, C.A. Ávila-Orta, A. Almendárez-Camarillo, J.G. Martínez-Colunga, E.Hernández-Hernández, V.J. Cruz-Delgado, P. González-Morones, S.G. Solís-Rosales and J.A. González-Calderón, "Non-isothermal crystallization behavior of isotactic polypropylene/copper nanocomposites. Journal of Thermal Analysis & Calorimetry," vol.143, Mar. 2021.
- [15] X.Q. Zhang, R.F. Shen, X.J. Guo, X. Yan, Y. Chen, J.T. Hu and W.Z. Lang, "Bimetallic Ag-Cu nanoparticles anchored on polypropylene (PP) nonwoven fabrics: Superb catalytic efficiency and stability in 4-nitrophenol reduction. Chemical Engineering Journal," vol.408, pp.128018, Mar. 2021, <https://doi.org/10.1016/j.cej.2020.128018>.
- [16] S. Lu, J. Wu, Y. Xia and C. Hu, "Effect of ionising irradiation on silver release from polyolefin/silver nanocomposite films into food simulants. Food Additives & Contaminants: Part A," vol.38, pp.681-690, Apr.2021, <https://doi.org/10.1080/19440049.2021.1878284>.
- [17] G.E. Ayas, Z. Ayhan, D. Duraccio, C. Silvestre and S. Cimmino, "Application of Polypropylene-Based Nanocomposite Films for Sliced Turkish Pastrami under Vacuum/Modified Atmosphere Packaging: A Pilot Study. Coatings," vol.10, pp. 1125, Nov. 2020, <https://doi.org/10.3390/coatings10111125>.
- [18] F. Zia, K.M Zia, S. Tabasum, M.K. Khosa and M. Zuber, "Preparation of hydroxyethyl cellulose/halloysite nanotubes graft polylactic acid-based polyurethane bionanocomposites. International journal of biological macromolecules," vol.153, pp.591-599, Jan. 2020, <https://doi.org/10.1016/j.ijbiomac.2020.03.038>.
- [19] E. Ananchaenwong, W.Chueangchayaphan, N.Rakkapao, S.Marthosa and B. Chaisrikhun, "Thermo-mechanical and antimicrobial properties of natural rubber-based polyurethane nanocomposites for biomedical applications. Polymer Bulletin," vol.78, pp. 833-848, Feb. 2021, <https://doi.org/10.1007/s00289-020-03137-z>.
- [20] L. Sisti, G. Totaro, A. Celli, L. Giorgini, S. Ligi and M. Vannini, "Bio-Based Furan-Polyesters/Graphene Nanocomposites Prepared by In Situ Polymerization. Polymers," vol. 13, pp. 1377, Jan.2021, <https://doi.org/10.3390/polym13091377>.
- [21] C. Arumugam, S. Arumugam and S. Muthusamy, "Mechanical, thermal and morphological properties of unsaturated polyester/chemically treated woven kenaf fiber/AgNPs@ PVA hybrid nanobiocomposites for automotive applications. Journal of Materials Research and Technology," vol.9, pp. 15298-15312, Nov.2020, <https://doi.org/10.1016/j.jmrt.2020.10.084>.
- [22] X. Song, Y. Wang, C. Jiao, M. Huang, G.H. Wang and H. Jiang, "Microstructure regulation of polyamide nanocomposite membrane by functional mesoporous polymer for high-efficiency desalination. Journal of Membrane Science," vol.1, pp. 117783. Mar. 2020, <https://doi.org/10.1016/j.memsci.2019.117783>.
- [23] A. Korde, B. Min, A. Ganesan, S. Yang, Z. Wang, A. Grosz, C.W. Jones and S. Nair, "AEL Zeolite Nanosheet-Polyamide Nanocomposite Membranes on α -Alumina Hollow Fibers with Enhanced Pervaporation Properties. Industrial & Engineering Chemistry Research," vol. 59, pp. 14789-14796, Jul.2020, <https://doi.org/10.1021/acs.iecr.0c02327>.
- [24] Y. Huang, B. Chen, Z. Lv and F. Guo, "Facile fabrication of durable super hydrophobic SiO₂/polyacrylate composite coatings with low nanoparticle filling. Journal of Coatings Technology and Research," vol.17, pp. 1289-1295, Sep. 2020, <https://doi.org/10.1007/s11998-020-00347-y>.
- [25] I.X. Yin, J. Zhang, J.S. Zhao, M.L. Mei, Q. Li and C.H. Chu, "The antibacterial mechanism of silver nanoparticles and its application in dentistry. International journal of nanomedicine," vol.15, pp. 2555, 2020, doi: 10.2147/IJN.S246764.

- [26] J. Compton, D. Thompson, D. Kranbuehl, S. Ohl, O. Gain, L. David and E. Espuche, "Hybrid films of polyimide containing in situ generated silver or palladium nanoparticles: effect of the particle precursor and of the processing conditions on the morphology and the gas permeability. *Polymer*," vol. 47, pp. 5303-13, Jul.2006. <https://doi.org/10.1016/j.polymer.2006.05.048>
- [27] S.S. Abdalla, H. Katas, F. Azmi and M.F. Busra, "Antibacterial and anti-biofilm biosynthesised silver and gold nanoparticles for medical applications: Mechanism of action, toxicity and current status. *Current drug delivery*," vol.17, pp. 88-100, Feb. 2020, <https://doi.org/10.2174/1567201817666191227094334>.
- [28] J.K. Sohal, A. Saraf and K.K. Shukla, "Silver Nanoparticles (AgNPs): Methods of Synthesis, Mechanism of Antimicrobial Action and Applications. *Multidisciplinary Research and Development*," vol.57, 2021.
- [29] S. Ali, X. Chen, M.A. Shah, M. Ali, M. Zareef, M. Arslan, S. Ahmad, T. Jiao, H. Li and Q. Chen, "The avenue of fruit wastes to worth for synthesis of silver and gold nanoparticles and their antimicrobial application against foodborne pathogens: A review. *Food Chemistry*," vol.20, pp. 129912, Apr. 2021, <https://doi.org/10.1016/j.foodchem.2021.129912>.
- [30] S. Dixit, G. Mishra and V. L. Yadav, "Optimization of novel bio-composite packaging film based on alkali-treated Hemp fiber/ polyethylene/polypropylene using response surface methodology approach. *Polymer Bulletin*," vol.13, pp. 1-25, Mar.2021, <https://doi.org/10.1007/s00289-021-03646-5>.
- [31] S. Lu, J. Wu, Y. Xia and C. Hu, "Effect of ionising irradiation on silver release from polyolefin/silver nanocomposite films into food simulants. *Food Additives & Contaminants: Part A*," vol. 38, pp. 681-690, Apr. 2021, <https://doi.org/10.1080/19440049.2021.1878284>.
- [32] J.H. Lee, D. Jeong and P. Kanmani, "Study on physical and mechanical properties of the biopolymer/silver based active nanocomposite films with antimicrobial activity. *Carbohydrate polymers*," vol. 224, pp. 115159, Nov.2019, <https://doi.org/10.1016/j.carbpol.2019.115159>.
- [33] S. Shankar and J.W. Rhim, "Preparation and characterization of agar/lignin/silver nanoparticles composite films with ultraviolet light barrier and antibacterial properties. *Food Hydrocolloids*," vol.71, pp. 76-84, Oct. 2017, <https://doi.org/10.1016/j.foodhyd.2017.05.002>.
- [34] S. Roy, S. Shankar and J.W. Rhim, "Melanin-mediated synthesis of silver nanoparticle and its use for the preparation of carrageenan-based antibacterial films. *Food Hydrocolloids*," vol.88, pp.237-46, Mar.2019, <https://doi.org/10.1016/j.foodhyd.2018.10.013>.
- [35] Y. Qin, Y. Liu, L. Yuan, H. Yong and J. Liu, "Preparation and characterization of antioxidant, antimicrobial and pH-sensitive films based on chitosan, silver nanoparticles and purple corn extract. *Food Hydrocolloids*," vol.96, pp.102-111, Nov.2019, <https://doi.org/10.1016/j.foodhyd.2019.05.017>.
- [36] S.J. Peighambari, S.H. Peighambari, N. Pournasir and P.M. Pakdel, "Properties of active starch-based films incorporating a combination of Ag, ZnO and CuO nanoparticles for potential use in food packaging applications. *Food packaging and shelf Life*," vol. 22, pp. 100420, Dec. 2019, <https://doi.org/10.1016/j.fpsl.2019.100420>.
- [37] S. Jafarzadeh, S.M. Jafari, A. Salehabadi, A.M. Nafchi, U.S. Kumar, H.A. Khalil, "Biodegradable green packaging with antimicrobial functions based on the bioactive compounds from tropical plants and their by-products. *Trends in Food Science & Technology*," vol.100, pp. 262-277, Jun. 2020, <https://doi.org/10.1016/j.tifs.2020.04.017>.
- [38] S.J. Peighambari, S. H. Peighambari, N. Pournasir and P.M. Pakdel, "Properties of active starch-based films incorporating a combination of Ag, ZnO and CuO nanoparticles for potential use in food packaging applications. *Food packaging and shelf Life*," vol.22, pp. 100420, Dec.2019, <https://doi.org/10.1016/j.fpsl.2019.100420>.
- [39] S. Dehghani, S.H. Peighambari, S.J. Peighambari, S.V. Hosseini and J.M. Regenstein, "Improved mechanical and antibacterial properties of active LDPE films prepared with combination of Ag, ZnO and CuO nanoparticles. *Food Packaging and Shelf Life*," vol. 22, pp. 100391, Dec. 2019, <https://doi.org/10.1016/j.fpsl.2019.100391>.
- [40] H. Hajizadeh, S.J. Peighambari, S.H. Peighambari and D. Peressini, "Physical, mechanical, and antibacterial characteristics of bio-nanocomposite films loaded with Ag-modified SiO₂ and TiO₂ nanoparticles. *Journal of food science*," vol. 85, pp. 1193-1202, Apr. 2020, <https://doi.org/10.1111/1750-3841.15079>.

10.22214/IJRASET

45.98

IMPACT FACTOR:
7.129

IMPACT FACTOR:
7.429

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Call : 08813907089 (24*7 Support on Whatsapp)