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Thermomechanical and Antioxidative Properties of *Monodora Myristica* Infused Polysulfone Active Film Package

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Abstract Natural antioxidant was extracted from *Monodora myristica*, a shrub grown majorly in South-East Nigeria, otherwise called "Ehuru" by the Ibo tribe in Nigeria. About 0 to 5% w/w compositions of *Monodora myristica* antioxidant extract (MAE) were infused into polysulfone (PS) resin. Another set of samples were compounded with 5% α-tocopherol (AT) in PS resin and other set of samples were infused with a combination of MAE and AT in PS resin. Tetrahydrofuran (THF) and *N*-methylpyrrolidinone (NMP) were used as solvents at the ratio 3:1. During the blending, mechanical, physico-chemical, thermal, morphological and barrier properties of the films were checked and compared with that of the pure PS. The antioxidative ability of the produced films was investigated using DPPH method. The results obtained show that tensile strength of the blended PS films reduced significantly with higher concentration of MAE accompanied with significant increase in elongation at break (EAB) when compared to that of the pure PS, while the antioxidative ability of the films increased significantly with addition of the MAE ($p < 0.05$). Results of the thermal property reveal that the glass transition temperature (T_g) for 5% MAE/AT active PS film was near 102.05°C higher than that of the pure PS. SEM micrographs showed a reduction in porosity of the pure PS films as the concentration of the MAE antioxidant increased from 1 to 5% w/w. Addition of 5% concentration MAE in PS produced very smooth and homogenous surface film without separation. The rate of permea-tion of oxygen gas into the active PS films reduced significantly from 0.1079±0.25 cm^3s^{-1} to 0.0277±0.17cm^3s^{-1} as the concentration of MAE in the film increased.

Keywords: *active polymer package, natural antioxidant, monodora myristica, spice extract, lipid preservation*

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1. Introduction

Packaging technology has these days, concentrated on packaging materials that have antioxidative qualities. This reduces lipid food deterioration caused by oxidation and microbial contamination, which are the highest means of lipid food spoilage. Antioxidative packaging systems containing active materials provide desirable preservative roles to food products. This process is termed "active packaging" because the package acts as an antioxidant amongst its other functions [1,2]. Active packaging is now common for the fact that many researchers have reported on the effect of directly adding natural antioxidants into bulk food, because flavonoids in most natural antioxidants acts as pro-oxidant to food. Furthermore, Samsudin et al., [3] and Tátraaljai, et al [4], reported that food preservation through the use of chemical substances have been responsible for some of health challenges in human. These challenges ranges from carcinogenic effects of butylated hydroxyl anisole (BHA) and butylated hydroxyl toluene (BHT) to interference of BHA in the human hormone (endocrine) system. These substances tend to migrate from the package into food especially during heating and long term storage. The natural antioxidants are considered safe since they belong to the component of food generally regarded as safe [5]. For these reasons therefore, it has become imperative to adopt active materials in food packaging.

1.1. Literature Review

Lipid oxidation is considered a principal means of deterioration or spoilage of oily foodstuffs such as vegetable oils, animal fats, flavours, nuts, processed meats and snack products [6,26]. The need for antioxidants is not limited to high-lipid foods, but also includes products such as cereals, which contain only 2 to 5% lipid components [7]. Oxidation not only negatively influences the chemical, sensory, flavour, texture and colour, and nutritional properties of edible oils and fatty foods, but also produces free radicals and reactive oxygen species (ROS), which have been reported to be associated with most health problems encountered in humans like carcinogenesis, inflammation, aging and cardiovascular disorders motility, chemotherapy response, and drug resistance [7]. Therefore, oxidation plays a key role in determining what a particular oil or fat can be used for and also its shelf-life [6,8,9].

Several works have been reported on the incorporation of natural antioxidants from leaf spice extracts as active agents incorporated into polymer food packaging. Extracts from green tea, oregano *(thyme)*, Rosemary (*Rosmarinus officinalis L.*), and *Camellia sineensis L.* are a few examples [10,11,12]. Other published articles dwelt on the incorporation of root spices such as Cucumin (*turmeric*) and Ginger *(Zingiber officinale Rosc.)* as active agents [13], while in some other publications , the mechanical properties, especially tensile strength (TS) and elongation at break of the active polymer films were greatly affected [14]. Polyvinyl alcohol active films incorporated with curcumin additive showed that tensile strength decreased with curcumin antioxidant, while the elongation at break increased. Similarly, Martinez-Pardo et al. [15] observed that α-tocopherol incorporated in thermoplastic-starch nano-hybrid film significantly decreased TS of chitosan films, while Siripatrawan and Harte, [16] reported that the incorporation of green tea extract in chitosan-based films caused a significant reduction in tensile strength when compared to that of pure chitosan-based films, without green tea extract . Noronha et al. *[17]* and Jongjareonrak et al. [25] also reported a significant TS decrease in fish skin gelatine films incorporated with α-tocopherol, and the elongation at break significantly decreased with the addition of α-tocopherol nanocapsules (NCs) when compared to that of pure PS films. However, as far as could be ascertained, no study on the use of locally sourced seed spice from the South-East part of Nigeria have been reported as active agent incorporated in polymer film packaging material. Therefore, this study investigated the thermo-mechanical property and antioxidant performance of the seed spice, *Monodora myristica*, otherwise popularly called *"Ehuru"* obtained from South-East Nigeria, as active additive in polymer film packaging material.

2. Materials and Methods

2.1. Materials

α-tocopherol of 98% purity, 2,2-Diphenyl-1- picrylhydrazyl (DPPH), anhydrous sodium carbonate, sodium thiosulphate, chloroform, PS resin, THF, NMP and methanol were purchased from Sigma Aldrich, (Pty), South Africa. *Monodora myristica* seed was locally sourced from a school farm in South-East Nigeria. All chemicals used for this study were of analytical grade and used without further pre-treatment.

2.2. Preparation of Monodora Myristica Extract

Monodora myristica extract was prepared according to Gurnani et al, [18] after modification. The collected *Monodora myristica* seeds were dried in an oven at 50°C until a constant weight was obtained and then crushed to fine powder using a laboratory mill and sieved to approximately 0.8 µm in size. About 100 g of the seed powder was sequentially extracted for 3 days with 500 mL of 70% ethanol, using a soxhlet apparatus in an oil bath at 70°C. The extracts obtained were filtered and concentrated at 70°C by using a rotary evaporator (Rotavapor R-200, BÜCHI Switzerland). The concentrate was further dried in hot air oven to give a dark brown gell, otherwise called the extract. The crude extract (MAE) was collected in an air tight container and refrigerated at 4±2°C until further analysis.

2.3. Preparation of Active Packaging Material

Three types of films were produced. One, containing different percentage compositions of only the *Monodora myristica* antioxidant extract (MAE), another containing α-tocopherol (AT) and the last containing a blend of α-tocopherol and MAE. MAE was blended with polysulfone (PS) resin at different percentage compositions of 0, 1, 2.5, 5% w/w. Another PS resin was blended with 5% standard natural antioxidant; α-tocopherol (AT) and another equal blend of 5% MAE/AT. Tetrahydrofuran (THF) and *N*-methylpyrrolidinone (NMP) were used as solvents in ratio 3:1. 10 g of PS was dissolved in the mixture of NMP and THF at room temperature. The mixture was agitated under high speed mixer for 24 hours until complete dissolution. The resulting solution was mixed with the varying compositions of MAE and α-tocopherol (AT). The films were produced by casting on a glass plate using a casting blade. The casting solution was kept at room temperature for at least 2 hours before casting to remove air bubbles. The newly cast was immediately immersed in distilled water coagulation bath within 10 sec to form the active film. After immersion, the residue solvent mixtures in the film were removed by immersing in distilled water for at least 24 hours. A pure PS film with 0% W/W of the fillers (MAE/AT) was prepared at the same condition for comparison.

2.4. Physico-chemical and Thermochemical Properties of the Pure PS Films

Images of the surface and cross section of the films were obtained by Scanning Electron Microscopy (SEM) to expose microstructure of the films and additive dispersion within the PS matrix. All samples were examined using an accelerating electron beam at a voltage of 25.0 kV at 5Kx magnification.

The permeation rate of the films were measured according to ASTM D3985 at 90 cm Hg pressure and operating temperature of 25°C while the average film thickness of 0.006cm using oxygen permeability tester: QT-OPT 300.

Fourier transform infrared spectroscopy (FTIR) was carried out in the sample to observe the structural interactions between the PS and the MAE or α- tocopherol. The films were conditioned at room temperature for 71 days in a desiccator containing silica gel. 0.6 to 0.8 mg of PS film samples were placed on the cell sensor surface clamped onto the mount of the spectrophotometer. Scanning was done in transmittance mode from 4000 cm^{-1} to 650 cm^{-1}, 16 scans per spectrum were recorded with a resolution of 4 cm^{-1}. The spectra obtained were used to determine possible interactions of functional groups between the PS molecular chains and the MAE and α-tocopherol (AT).

The tensile strength (TS) and elongation at break (EAB) of the active film were determined at room temperature by using a TA.XT2 Stable Microsystems (SMD, England), according to ASTM standard method D882. The TA.XT2 was equipped with 7.5 kg static load cell. The films were cut into equal rectangular shapes of 10 cm length x 2.5 cm width and mounted between the grips of the TA.XT2 instrument. The initial grip spacing and crosshead speed were set at 30 mm and 25 mmmin-1 respectively. The tensile strength was expressed as maximum force at break (FMax) divided by the initial cross-sectional area (A) of the sample film (see Equation 2).

$$TS = \frac{F_{\max}}{A}. \quad (2)$$

The elongation at break (EAB) was expressed as the percentage of the original length at break as presented in equation (3).

$$EAB(\%) = \frac{L}{L_o} x100 \quad (3)$$

TS and the EAB were expressed in MPa and percentage (%), respectively. Values were mean of replicated three measurements for each film sample to ensure accuracy. Thickness of the films were measured by using micrometre screw gauge (MDC-25S, Japan).

Thermal analysis was carried out on the neat PS and active PS films using the Perkin-Elmer Differential Scanning Calorimeter (DSC). 5 to 8 mg of the PS active polymer films were measured by using a Differential Scanning Calorimeter (DSC) at the heating rate of 20°Cmin^{-1} from 0 to 340°C. The glass transition temperature (T_g), and melting temperature (T_m) of the active polymer films was determined from the DSC curves [19].

Antioxidant effect of the active film was determined by using the method as described by Jouki *et al.*, [20]. 0.1 g of the active polymer films was cut into small pieces and mixed with 2 mL of methanol. The mixture was vigorously shaken in a vortex for 3 minutes and allowed to stand at room temperature for 3 hours and centrifuged at 2300 rpm for 10 min. The supernatant was analysed for DPPH radical scavenging activity. An aliquot of methanol extract (1 mL) was mixed with 2 mL of 0.1 mM DPPH in methanol. The mixture was vigorously shaken in a vortex for 1 min and allowed to stand at room temperature in the dark for 30 min. The absorbance was measured at 517 nm using a UV- VIS spectrometer. The methanol was used as control and was mixed with 0.12 mM DPPH. DPPH radical scavenging activity was calculated with equation 1. [21];

$$Radical\ scavenging\ activity(\%) = \frac{A_{Reference} - A_{Sample}}{A_{Reference}} \times 100 \quad (1)$$

Where; A_{Sample} is the absorbance of sample and $A_{Reference}$ is the absorbance of the DPPH solution without the sample film.

3. Results and Discussion

Table 1 shows the measured properties; TS (MPa), EAB (%), antioxidative property (%) and permeation rate (cm^3s^{-1}), of the PS active films infused with 0.0% MAE (Neat PS), 1.0% MAE, 2.5% MAE, 5.0% MAE, 5.0% MAE, 5.0% MAE, 5.0% AT, and a blend of 5.0% MAE and 5%AT. Figure 1 – Figure 3 show clearly the effect of MAE concentration in PS active films on the TS, EAB, antioxidative property and permeation rate of the film.

3.1. Mechanical Property of the Active Films

A good flexibility to prevent breaking during food packaging is very important for polymer films for food packaging. From Table 1, the effect of concentration of *Monodora myristica* antioxidant extract and AT blend on the tensile strength and elongation at break show that there is a significant decrease in tensile strength of the films incorporated with either MAE or AT compared to that of pure PS film without any antioxidant ($p < 0.05$). Figure 1 show the comparison of line plot between TS and EAB on 0 to 5% concentration of MAE in PS active film.

Table 1. Mechanical, antioxidant and barrier properties of the PS active films

PS Property	0.0% MAE (Pure PS)	1.0% MAE	2.5% MAE	5.0% MAE	5.0% AT	5.0% MAE and 5% AT
TS (MPa)	8.132±0.31	7.426±0.34	7.538±0.25	7.739±0.71	5.636±0.22	6.405±0.54
EAB (%)	2.002±0.72	2.353±0.11	2.673±0.33	3.221±0.09	1.702±0.55	1.751±0.23
Antioxidative property (%)	3.100±0.10	10.077±0.47	22.590±0.24	48.097±0.70	51.587±0.26	40.901±0.67
Permeation rate (cm^3s^{-1})	0.1079±0.25	0.0463±0.34	0.0593±0.11	0.0277±0.17	0.2209±0.41	0.1079±0.15

TS = Tensile strength, EAB = Elongation at Break, MAE = Monodora myristica extract, AT = α-tocopherol.

Figure 1 show that the percentage elongation at break values of the neat PS was found to be 2.002±0.72%. However, the values of EAB for the MAE incorporated PS films ($p<0.05$) significantly increased as concentration of the MAE increased from 0 to 5%. This effect is more pronounced in films containing AT and MAE-AT blend films, which displayed significantly reduced EAB values of 1.702±0.55% and 1.751±0.23%, respectively ($p<0.05$). To avoid breaking during processing and use, films for packaging require high flexibility [22]. The pure PS gave the highest value of 8.132±0.31 MPa for tensile stress, while PS active films infused with MAE had an increase in their tensile stress from 7.426±0.34 MPa to 7.739±0.71 MPa as the percentage composition increased from 0 to 5%. Also, the addition of the same quantity of 5% AT showed very pronounced reduction in tensile stress compared to all concentrations of MAE investigated with value of 5.636±0.22 MPa. Figure 2 is a comparative chart of tensile strength of the films.

From Figure 2, the blending of the 5.0% MAE and AT obviously improved the tensile strength from 5.636±0.22MPa to 6.405±0.54MPa. This indicates that MAE active PS film gives a stronger film than AT and MAE/AT active PS film. These flexible characteristics of the MAE infused film may have been caused by the hydrophilic components present in the *Monodora myristica*, which also suggests that the plasticizing effect of the MAE could have caused intermolecular interactions between the extract and PS making the film relatively flexible. On the other hand, AT films may have behaved differently because of its hydrophobic nature. A homogeneous dispersion of hydrophobic AT into polymeric matrix of PS would increase the space between macromolecule chains, this would reduce ionic and hydrogen bonding between the chains and induce the development of structural discontinuities in the films [17]. The table also shows that the antioxidant level of the active films increased as concentration of the MAE incorporated increased while the permeation rate reduced as concentration of the MAE added increased.

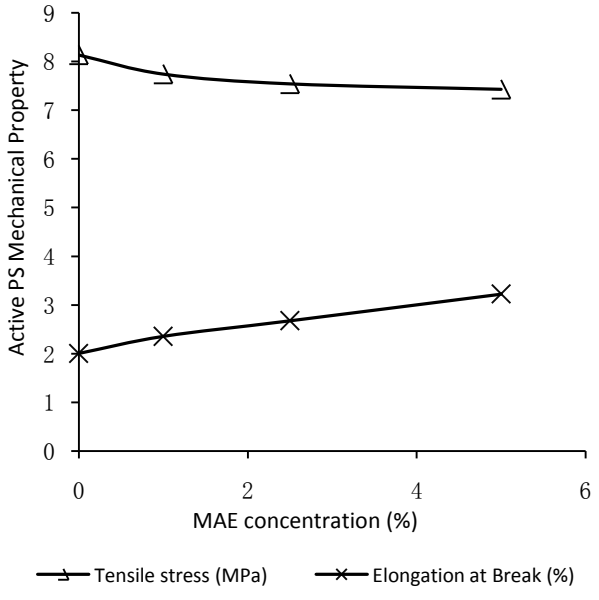

Figure 1. PS mechanical properties vs MAE concentration from 0 - 5%

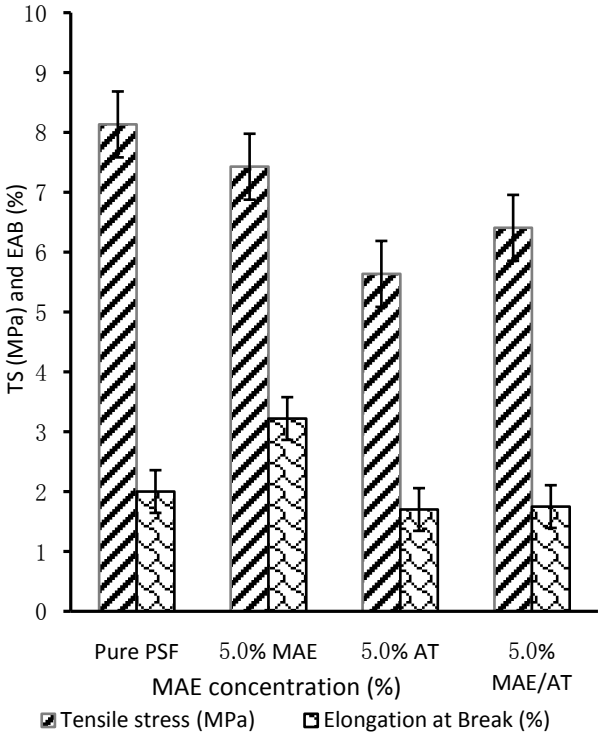

Figure 2. Comparative mechanical properties of the active films

3.2. Results of Antioxidation Tests of PSF Active Films

A plot of the antioxidative properties of the pure and infused active PS films against percentage of MAE concentration in the PS films is presented in Figure 3.

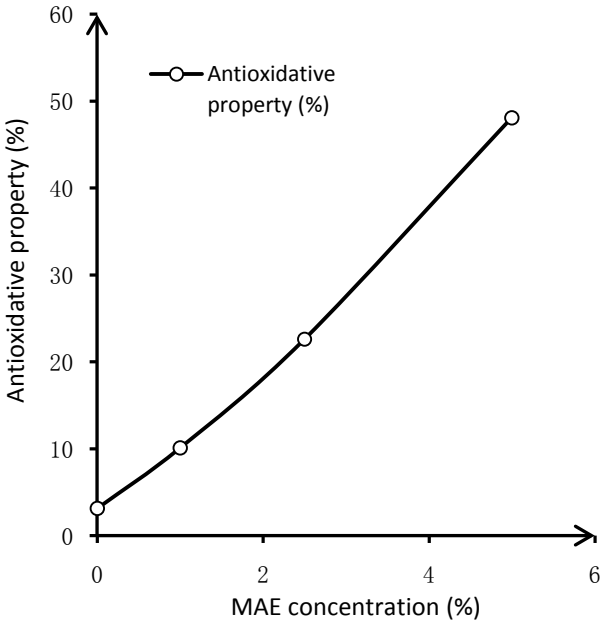

Figure 3. Antioxidative property of active PS films vs MAE concentrations

The antioxidative property of the films increased tremendously from 3.100±0.10% for the pure PS films to 48.097±0.70% for 5% MAE active PS film. The 5% AT active PS film had 51.587±0.26% antioxidative property. However, the MAE/AT blend had a reduced antioxidative

property of 40.901±0.67%, which may be attributed to an overshadowing effect of MAE antioxidant over AT antioxidant.

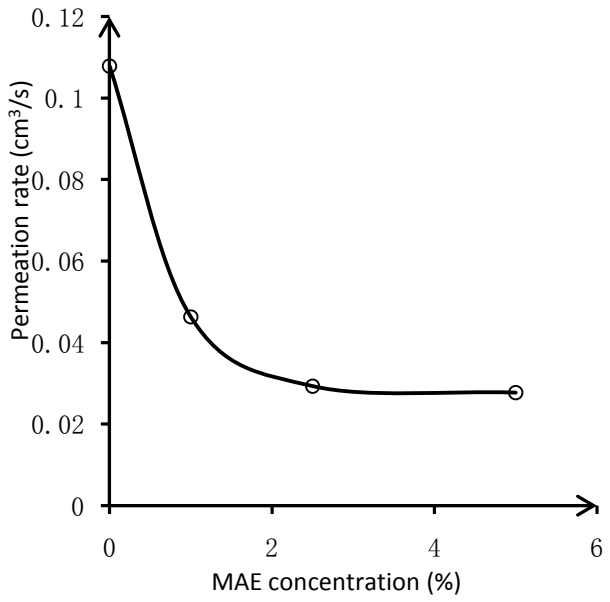

Figure 4. Permeation rate against MAE concentration

3.3. Permeation Rate of Active PS Films

Results of barrier properties in Table 1, are plotted in Figure 4, where the permeation rate in cubic centimeters per second (cm^3s^{-1}) is plotted against concentration of MAE in PS active film, 0 to 5% w/w. Interestingly, the permeation rate could be an indication of the nature of porosity in the PS films as a result of inclusion of MAE, AT and a blend of MAE and AT in the active PS films.

From Figure 4, as the–concentration of MAE in the active PS films increased, there was a downward slide of the permeation rate showing drastic reduction in permeation rate. The MAE active PS films had lower permeation rate, hence lower porosity compared to the pure PS film.

3.4. Thermal Property of Active PS Films

Table 2 shows the thermal transitions of the active PS films by differential scanning calorimetry (DSC).

Table 2. DSC Thermal Properties of the Active Films

Samples	1st Onset temp, T_o (°C)	T_g (°C)	T_c(°C)	T_m(°C)	Degradation Temp (°C)
0% MAE	62.09	87.5	192.6	230.1	255
5% MAE	62.01	87	153	210	279.5
5%AT	63.85	87.08	167	247	284.5
5% MAE and 5% AT blend	62.01	102.05	184.5	237	257.6

The results revealed that all the active PS film samples have marginal differences in their glass transition temperature (T_g) of around 87°C apart from that with 5% MAE/AT, which has T_g of 102.05°C. The T_g is the temperature at structural transition of polymer material changes from an amorphous solid or glassy state to a more viscous rubbery state. Below T_g of 87 ±0.1°C, the PS films are rigid and brittle whereas above T_g, films become flexible and pliable.

The table also shows the melting point of the pure PS to be at 230.1°C. However, its MAE active PSF film had a reduced melting point temperature of 210°C. On the other hand, AT active PS film recorded a higher melting temperature of 247°C, the reason being that AT is a heat-stable phenolic compound and it is in agreement with the report of Siró et al., [23]. However, its blend, MAE and AT, had a reduced melting temperature of 237°C. The addition of either MAE or AT increased the degradation temperature significantly. However, the blend experienced a reduction in the degradation temperature, from the temperature of 284.5°C to 257.6°C. This shows that MAE is not a heat-stable compound compared to AT. The addition of the antioxidant MAE, AT or the blend to the film significantly reduced the crystallisation temperature (T_c) of the pure PS film from the temperature of 192.6°C to 153°C for 5% MAE active PS film, 167°C for 5% AT active PS film and 184.5°C for a blend of 5% MAE and 5% AT active PS films, respectively.

3.3. Fourier Transform Infrared (FTIR) Spectroscopy Analysis of the PS Active Films

The FT-IR spectra of the pure PS, MAE/PS and AT/PS active films are presented in Figure 5, Figure 6, and Figure 7 respectively.

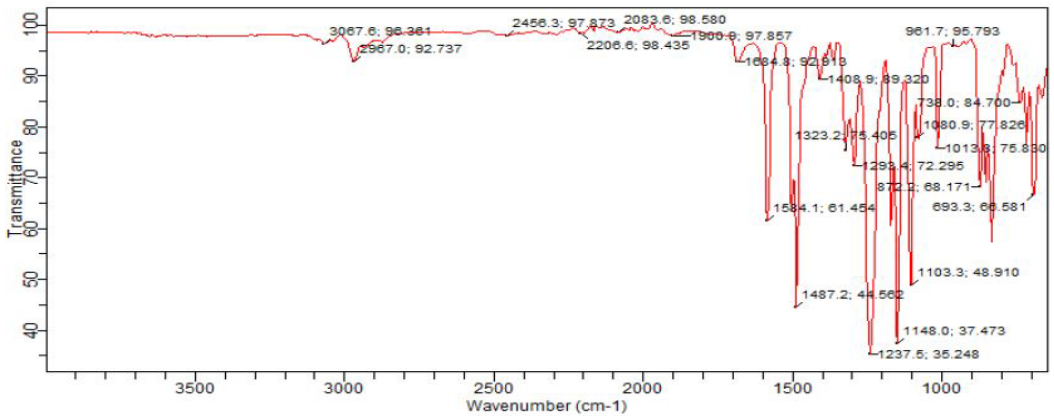

Figure 5. FTIR spectrum of pure MAE/PS compositions

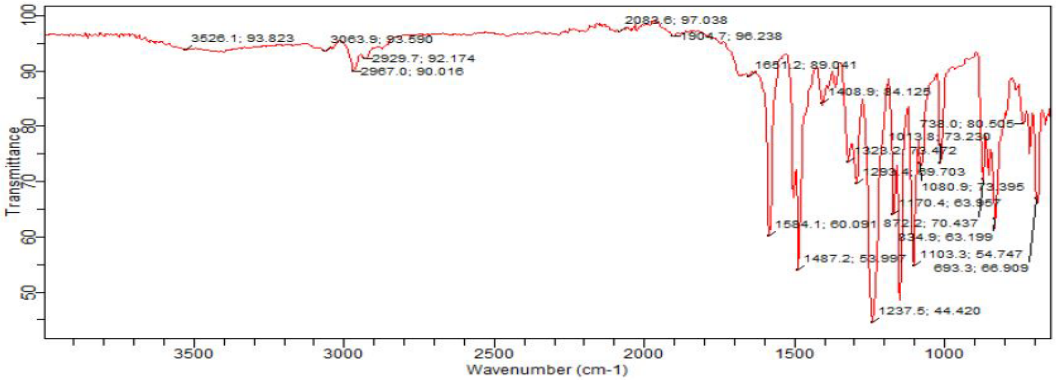

Figure 6. FTIR spectrum of 5% MAE / PS compositions

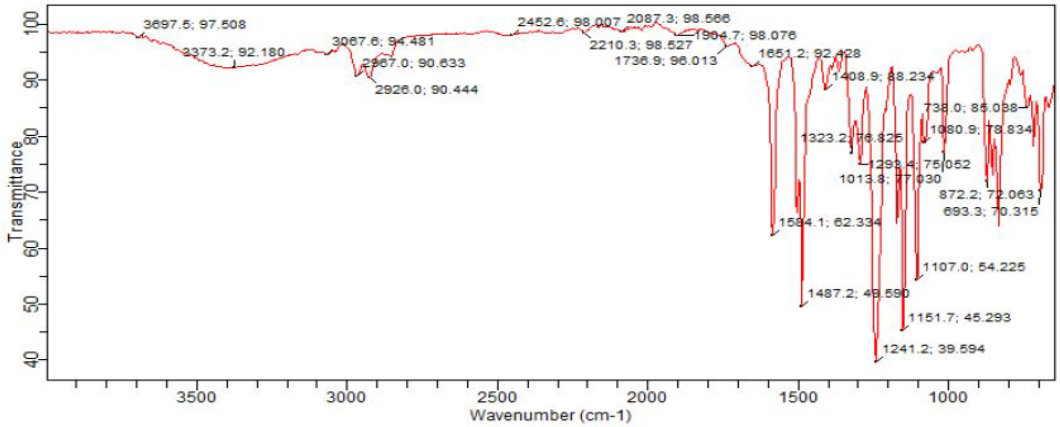

Figure 7. FTIR spectrum of 5% MAE/AT PS blend

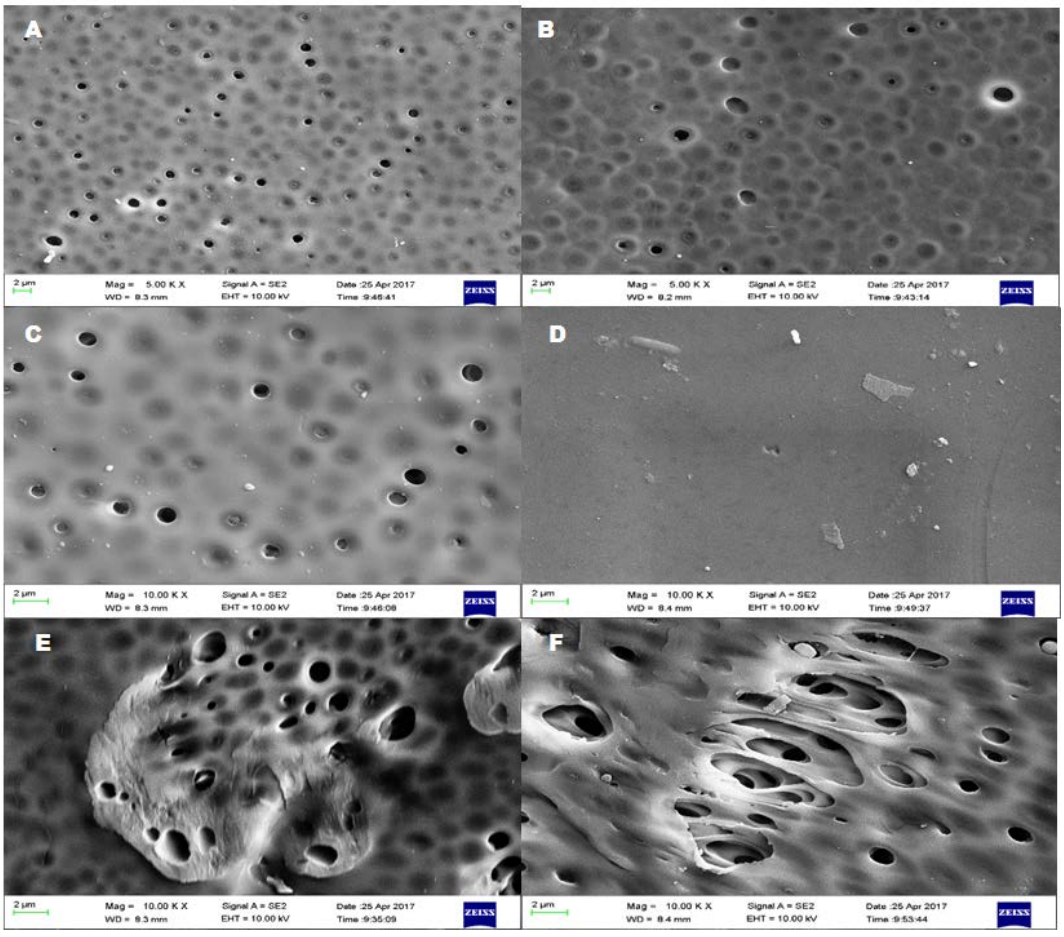

Figure 8. SEM Micrograph of the Active PS Films (**A:** Pure 0% MAE, **B:** 1% MAE, **C:** 2.5% MAE, **D:** 5% MAE, **E:** 5% blend of MAE/AT, **F:** 5% AT)

From the FTIR spectra shown in Figure 5 - Figure 7, the vibrations associated with the functional groups are not intense except in the fingerprint area between 400 cm^{-1} to 1500 cm^{-1} showing presence of less polar compounds which represent sp^2 aromatic C-H bending vibrations. Figure 5 represents spectra for the pure PS, where apart from the fundamental vibrations, there are no absorption bands beyond 3063.9 cm^{-1} for C-H bending. However, Figures 6 and 7 have shown additional peaks beyond 3063 cm^{-1} though very weak, which may be attributed to functional groups associated with MAE or AT additive in the PS active film. Whereas the pure PS has no antioxidant, therefore showed the fingerprint of PS without that of the antioxidants, the additional peaks in the active PS films should confirm the active components present in MAE and AT. From Figure 6, the peak at 1725 cm^{-1} and 1750 cm^{-1}, there is a weak overtone of carbonyl (-C=O) IR stretch vibrations attributable to an ester, ketone or aldehyde group in the MAE. Beyond 3063 cm^{-1}, the additional peak values and their associated functional groups are those at 3373.2 cm^{-1}, that may be associated with broad weak one-band secondary amine (-CO-NH_2) from acetamide ($CH_3C(=O)$-NH_2), which usually appeared the absorption band of 3400 cm^{-1}. There may also be the presence of N-H and N-H_2 stretching within the range of 3526.1 to 3697.5 cm^{-1}. Furthermore, from the other peak beyond 3063cm^{-1} is that at 3697.5 cm^{-1} for 5% MAE and 5% AT PSF compositions. This peak may be associated with hydroxyl (-OH) group from AT.

3.4. Morphology of the MAE Infused Active PS Film

Scanning electron micrographs of the active PS films in addition to that of the pure PS film are shown in Figure 8. The micrographs exposed the morphology of the films.

The micrograph of the pure PS film as shown in Figure 8A revealed a dense, grainy and tiny porous surface. The pores reduced as concentration of the MAE additive increased from 0 to 5% w/w, hence, as observed in Figures 8; B, C and D.

Incorporation of 5% w/w concentration of MAE, produced very smooth and homogenous surface film without apparent phase separation as shown by the compactness and no optical pores noticed in Figure 8D. This observation could be as a result of cross linking between the components of the MAE and the PS that resulted in reinforcement, which led to the sealing process. Similar observation of grainy and porous surface were noticed on PS compounded with only AT and blend of MAE and AT as shown in Figures 8E and F. This observation was more pronounce with only AT as additive as seen in Figure 8F, which showed larger porous structure in comparison to MAE or the blend. The addition of the MAE and AT blend or only AT to the PS, produced poor dispersion causing discontinuous phase within the polymer matrix. It showed that MAE significantly influenced the morphology of the PS structure. On the other hand, the addition of only AT could have caused an increased movement of the macromolecules in the film matrix molecules, which altered the PS morphology leading to weakened PS active films as shown in Figures 8 E and F. This similar morphological change was observed by Arrieta et al., [22] and Arrieta et al., [6] when a phenolic compound D-limonene (plasticizer) was incorporated in PLA polymer. Pedrielli *et al.*, [24] have earlier reported that flavonoids are more hydrophilic than α- tocopherol. These results show that the addition of hydrophobic substances modified interactions with PS polymer compound thereby weakening the film and altering the film morphology. While hydrophilic substances like the MAE would migrate to the voids observed in PS thereby acting as plasticizer and causing a seal because of their small nature.

4. Conclusion

This study has shown that active PS film can be produced using *Monodora myristica* (Ehuru) antioxidant seed extract (MAE). From the microstructure of the films, the infusion of 5% w/w concentration of MAE in the PS films produced compact, smooth and homogenous surface structure without apparent phase separation compared to the AT infused films and a blend of MAE and AT infused films which produced porous films susceptible to increased oxygen permeation rate. The tensile strength and thermal properties of the PS films were significantly improved by the addition of MAE active compound. The tensile strength of the MAE infused PS film was threaded off with an increase in elongation at break of the same film compared to those of pure PS film and AT, MAE and AT infused films, which may be as a result of the compactness of the MAE infused films on one hand, and increased porosity of the AT infused films . On the other hand, this translated to improved flexibility of the MAE active package film. The antioxidative property of the films increased as percentage composition of the MAE infused increased from 0 to 5%. The incorporation of MAE active component in the PS film presented a potential antioxidant ability, which could be used to enhance the scavenging ability of polysulfone film. Therefore, these films could be an alternative active polymer film for lipid food package.

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Conflict of Interest

There is no conflict of interest associated with part or all of this work.

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