





## Characterization of an eco-friendly active packaging film for food with ultraviolet light blocking ability

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### Abstract

An eco-friendly active packaging film for food with ultraviolet (UV) light blocking ability was prepared using nano-magnesium oxide (MgO), nano-zinc oxide (ZnO), nano-cellulose (NCC), and poly(lactic acid) (PLA). The results revealed that the four nanomaterials were evenly dispersed in the PLA films, but no chemical bonds formed according to infrared spectroscopy and scanning electron microscopy. Compared with other PLA films, the PLA films with ZnO were endowed with excellent UV absorption and its surface hydrophilicity was decreased. On the contrary, the PLA films with MgO, ZnO, and NCC had improved mechanical strength, better antimicrobial activity, lower oxygen permeability (OP), and water vapor permeability (WVP). The PLA film with nanoparticles is an excellent active packaging material with improved physical, mechanical, and barrier properties, which can also avoid the damage of food or active ingredients in packaging from UV radiation, and has a broad application prospect for the preparation of multilayered composite active packaging materials for food.

**Keywords:** eco-friendly; active packaging film for food; nano-powders; ultraviolet light blocking.

**Practical Application:** The application of films in food packaging is considered to be very promising. In this study, an eco-friendly active packaging film with ultraviolet light blocking ability was prepared using nano-magnesium oxide (MgO), nano-zinc oxide (ZnO), nano-cellulose (NCC) and poly(lactic acid) (PLA). Compared with other PLA films, the PLA films with ZnO was endowed with excellent ultraviolet absorption, and its surface hydrophilicity was decreased. On the other hand, the PLA film with MgO, ZnO, and NCC had improved mechanical strength, better antimicrobial activity, lower oxygen permeability (OP) and water vapor permeability (WVP). So, an eco-friendly active packaging film with ultraviolet light blocking ability has a broad application prospect for the preparation of multi-layer composite active packaging materials for food.

## 1 INTRODUCTION

With increasing concern about environmental pollution by petroleum-based plastics, biodegradable materials have attracted increasing attention of researchers. At present, biodegradable materials used in active food packaging include poly(lactic acid) (PLA) (Altan et al., 2018), poly( $\epsilon$ -caprolactone) (PCL) (Wang et al., 2019), poly(butylene succinate) (PBS) (Vorawongsagul et al., 2021), poly(hydroxyalkanoate) (PHA), poly(ethylene glycol) (PEG) (Hou-Yong et al., 2019), starch (Baek et al., 2019), cellulose derivatives (Kousheh et al., 2020), plant protein (soy protein isolate, zein, and wheat gluten) (Chavoshizadeh et al., 2020; Li et al., 2021; Lu et al., 2020), and animal protein (gelatin, keratin, and whey protein isolate) (Mohammadi et al., 2020; Nur Amila Najwa et al., 2020; Tinoco et al., 2020). PLA is considered the most feasible substitute for traditional plastic packaging because of its moderate barrier properties, high

crystal transparency, and good processability. However, its poor brittleness and other properties limit its application in food packaging (Rasal & Hirt, 2009).

At present, a series of natural active ingredients (antioxidants and antimicrobials) were added to packaging materials to prepare active and intelligent food packaging (Chavoshizadeh et al., 2020; Guo et al., 2020; Janani et al., 2020; Kong et al., 2020; Sharma et al., 2021). However, the stability of active ingredients to ultraviolet (UV) light, temperature, and oxygen are important for the active films in practical application (Vilela et al., 2017; Zhang et al., 2019). Especially, UV light showed a significant influence on the stability of natural active ingredients over other factors. Therefore, it is essential to improve the stability of natural active ingredients (Mohr et al., 2019; Yang et al., 2021; Yuan et al., 2019).

Currently, encapsulation technology (pickering emulsions, hydrogels,  $\beta$ -cyclodextrin, and microfiber) (Chen et al., 2019;

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Dammak et al., 2019), multilayer technology (Biswal & Saha, 2019; Konuk Takma & Korel, 2019; Oudjedi et al., 2019), or both (Estevez-Areco et al., 2020; Li et al., 2020; Yang et al., 2021) are used to protect the active ingredients in packaging material from environmental factors. Encapsulation uses tiny physical structure to protect the active substances directly, while the multilayer technology uses one or more layers of composite membranes to protect the active substances indirectly. Composite membranes generally include multiple active layers and protective layers. The protective layer must have some good characteristics, such as a high barrier, good mechanical properties, high chemical resistance, and UV protection (Yang et al., 2021).

To match the above requirements, a series of protective layers based on PLA and nanoparticles were prepared in this test. The application of nanoparticles in active packaging is considered to be very promising. Nanoparticles are incorporated into food-contact polymers to enhance mechanical and barrier properties. In addition, they are also effective antimicrobial agents and UV absorber. Currently, nanomaterials such as Ag<sup>+</sup> (Nur Amila Najwa et al., 2020; Yalcinkaya et al., 2017), CuO (Peighambardoust et al., 2019), nano-zinc oxide (ZnO) (Sun et al., 2020; Yadav et al., 2021), nano-magnesium oxide (MgO) (Swaroop & Shukla, 2018), and TiO<sub>2</sub> (Riahi et al., 2021) are used in active and intelligent packaging. They are generally recognized as safe compound in food industry approved by the Food and Drug Administration.

In this study, a variety of nanoparticles were used in the preparation of PLA active packaging materials. The aims of this study are to enhance the barrier and mechanical properties of PLA film and to give it excellent UV absorption capacity. Then, based on the PLA film, a variety of composite active packaging materials can be developed in the future, and the stability of active ingredients can be effectively guaranteed.

## 2 MATERIALS AND METHODS

### 2.1 Reagents and samples

PLA (code 4032D, density = 1.24 g/cm<sup>3</sup>, and  $M_w = 2.1 \times 10^5$  (g/mol)) was purchased from Nature Works LLC (Blair, Nebraska, USA). Trichloromethane, ZnO ( $\leq 100$  nm), MgO (50 nm), and cellulose (250 G) were purchased from Sigma (Nanjing, China).

### 2.2 Preparation of nano-modified PLA film

First, the PLA and nanomaterials (MgO, ZnO, and cellulose) were dried at 60°C for 24 h. Subsequently, 4 g of PLA and 100 mL of trichloromethane were added to a beaker and magnetic stirring was carried out for about 3 h until the PLA was completely dissolved. Next, nanomaterials (MgO, ZnO, nano-cellulose (NCC), and MgO/ZnO, with 1, 2, 3, and 4 g/100 g) were added to the PLA solution, and then the PLA solution was subjected to ultrasound for 15 min at 40 kHz and was stirred for about 1 h. The PLA solution was then coated onto a clean glass plate at room temperature and was allowed to naturally evaporate for 2–3 h to form a film. Finally, all films were stored at 45 °C for 24 h (Swaroop & Shukla, 2018).

### 2.3 Thickness

A total of five points were randomly selected on a film and the thickness of the film was measured using a micrometer (Mitutoyo, Kawasaki, Japan). Each sample had five parallels and we obtained the average value.

### 2.4 Transmittance

Each film was cut into a rectangle of 2 × 4 cm and the transmittance was measured using an UV–visible spectrophotometer (Mettler Toledo, Zurich, Switzerland). The wavelength range was set to 200–800 nm and each sample was taken in five parallels (Arrieta et al., 2015).

### 2.5 Water vapor permeability and oxygen permeability

First, each film was cut into a circle having a diameter of 6 cm and then 15 mL of water was added to each test cup (4.5 cm in diameter and 3 cm in height) to maintain the humidity at 90%. The membrane was fixed to the mouth of the cup with paraffin. The test cup was placed in a desiccator (relative humidity = 90%), weighed after 2 h and then taken out once every 3 h for weighing. Weight loss from each cup was measured as a function of time for 12 h. The test was done in duplicate and the mean value was reported.

The VAC-V1 OP tester (Industrial Physics, Boston, USA) was used in the experiment. The sample was stored at 25°C and 50% RH for 2 h and then cut into a circle having a diameter of 9.7 cm. The test piece was placed in a tester with a test area of 38.46 cm<sup>2</sup> and the test time was 8 h at an oxygen pressure of 0.5 MPa.

### 2.6 Tensile properties

TA-XT plus texture analyzer (Stable Micro System Ltd., Godalming, UK) was used in the experiment. The A/TG stretching die was selected and calibrated with a 5 kg weight. The initial distance of the holder was set to 50 mm, the test speed was fixed at 1 mm/s and the data were processed using Texture Exponent 32. The tensile strength (TS), modulus of elasticity (EM), and elongation at break (EAB) were calculated by using a stress curve. Each sample was tested eight times and four parallel samples were taken per sample for a sample size of 10 mm × 150 mm.

### 2.7 Scanning electron microscopy

An SEM FEI Quanta 200FEG (Hillsboro, OR, USA) was used to characterize the surface structure of PLA films, and the surface was coated with Au/Pd alloy before the measurement, using an E5 150 SEM coater (Polaron Equipment Ltd., Doylestown, PA, USA). The pressure was set to 10 kV (Dashiour et al., 2015).

### 2.8 Statistical method

Statistical analyses were carried out with ANOVA using IBM SPSS Statistics Version 23.0 and the differences between the trials were detected using the LSD test ( $P < 0.05$ ).

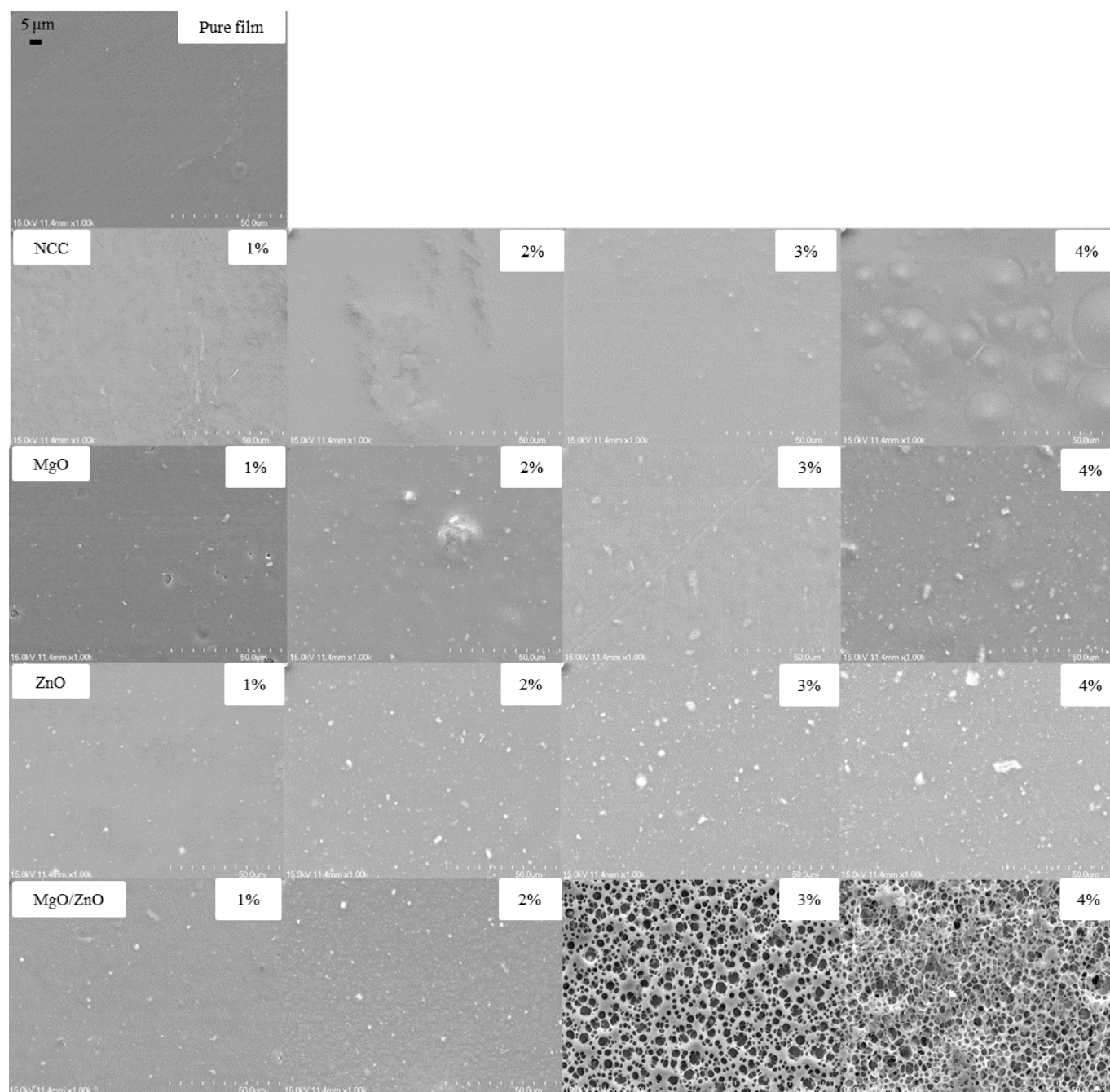
### 3 RESULTS AND DISCUSSION

#### 3.1 Characterization of nanomaterial-modified PLA films

##### 3.1.1 Scanning electron microscopy

An SEM image of the film surface is shown in Figure 1. The results showed that the surface of pure PLA film, 1% and 2% NCC films, was smooth and uniform and had no obvious aggregation of particles, while the 3% NCC film showed obvious aggregation of particles. The 4% NCC film had obvious convexity, which might be caused by the aggregation of NCC (Sun et al., 2020; Yadav et al., 2021). Similarly, the

surface of the film with 1–4% MgO and ZnO showed some granular bulges, which increased with the addition of MgO and ZnO. The MgO/ZnO films with different additions had significant differences, with 1% being the most uniform and 2% having some bumps and concave areas. The 3 and 4% showed discontinuous surfaces and uniform holes but nano-agglomerated particles were still uniformly dispersed between the holes. In addition, there was no significant difference between 1, 2, 3, and 4% films in terms of tensile and barrier properties. The pores on the PLA membrane surface may be caused by the different surface tensions of the two kinds of nanomolecules.



**Figure 1.** SEM images of various PLA films.



### 3.1.2 Transmittance

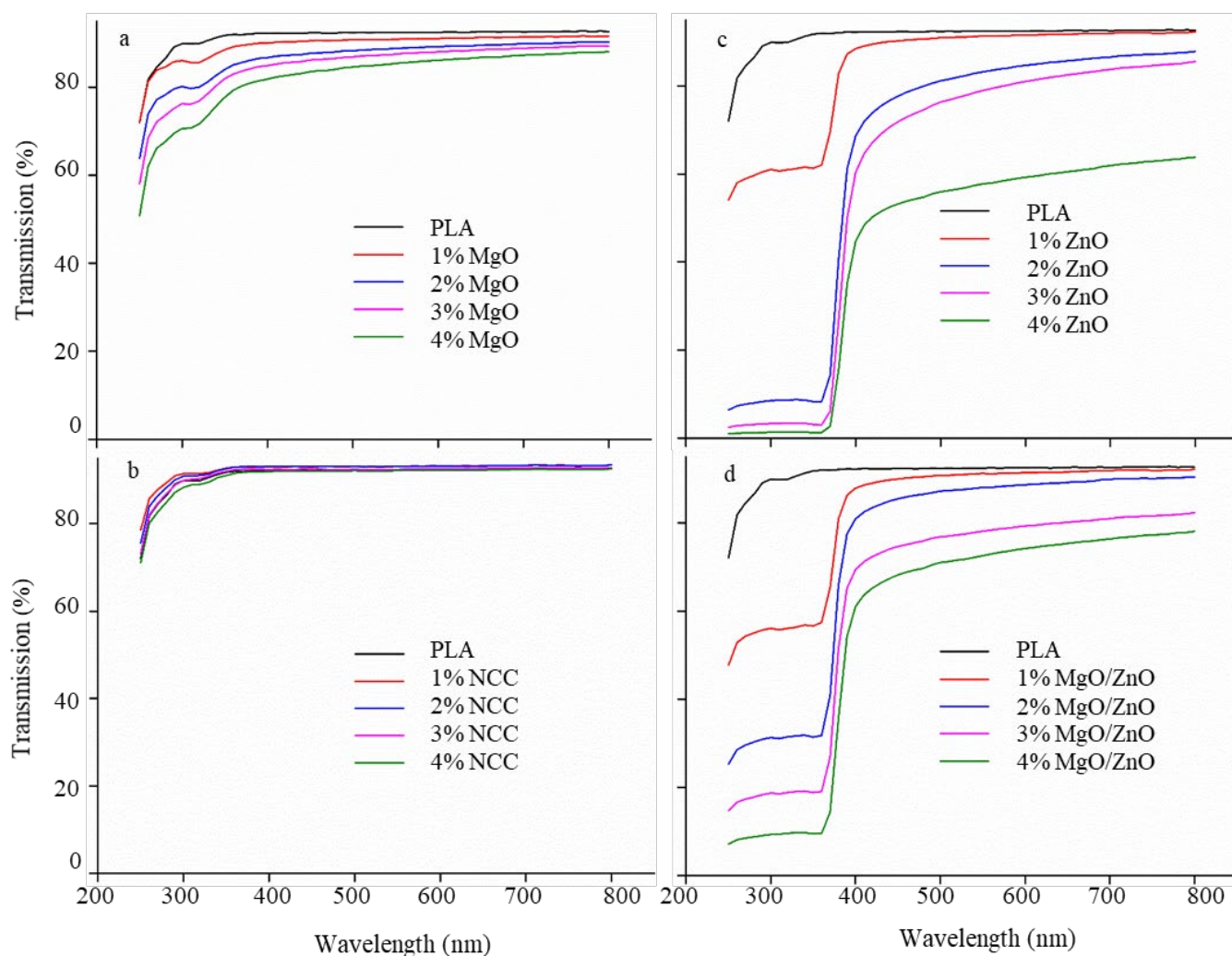
The transmittance of each PLA film was affected by adding nanomaterials during the processing. The effect of different groups on the transmittance is shown in Figure 2. Compared with the pure PLA films, the PLA-NCC film had no significant change. The transmittance of PLA/MgO, PLA/ZnO, and PLA/MgO/ZnO films decreased, indicating that ZnO and MgO could reduce the light transmittance of the PLA film. The transmittance of PLA/ZnO and PLA/MgO/ZnO films in the UV spectral region (200–400 nm) decreased significantly ( $P < 0.05$ ); the larger the amount of nanomaterials added, the lower the transmittance (Jiang et al., 2018). However, the light transmittance of PLA/MgO film in the UV spectral region did not decrease significantly, indicating that ZnO has a strong UV absorption effect. Compared with the control, the transmittance of 2% PLA/ZnO was lower than 10% and it could absorb most of the UV rays. Furthermore, there was no significant difference between 2, 3, and 4% PLA/ZnO films. This indicates that adding 2% ZnO to the film could achieve a good UV light absorption effect. Therefore, ZnO

has potential application in the packaging of UV-sensitive foods (Marra et al., 2016).

### 3.2 Tensile properties of the nano-modified PLA films

Mechanical strength is very important to the application of biodegradable materials in food packaging. Therefore, it is very important to study the mechanical properties of packaging film (Wen, et al., 2017). The main mechanical properties of PLA films such as TS, EM, and EAB are shown in Figure 3.

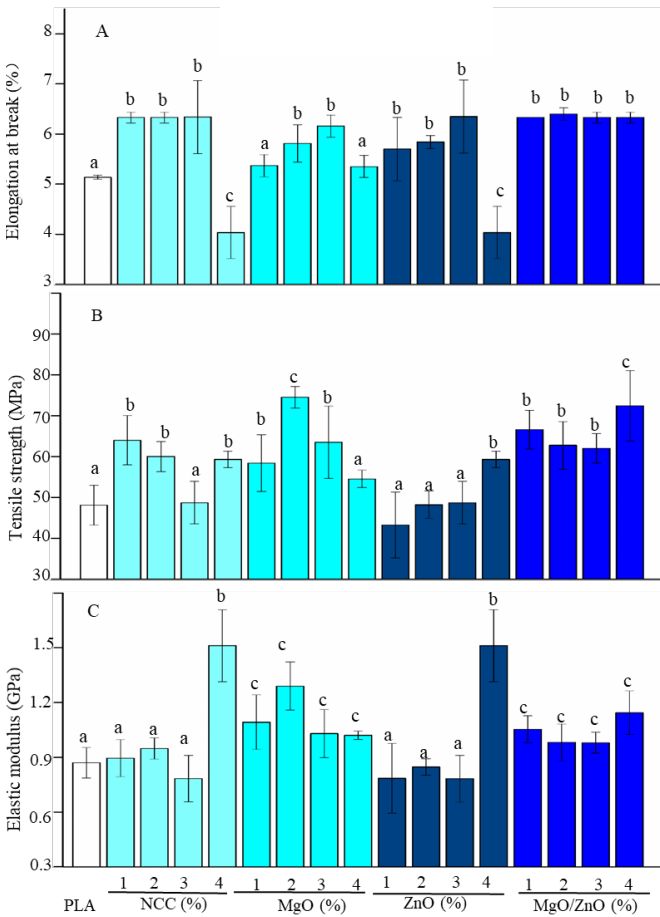
As shown in Figure 3, the mechanical properties of the nano-modified PLA film were significantly improved compared with the pure PLA film. TS, EM, and EAB of PLA films were 48.12 MPa, 0.87 GPa, and 5.13%, respectively. The results show that the TS and EAB of 1% NCC increased significantly by 31 and 23%, respectively, but its mechanical properties did not continue to improve with the increase in the added amount. TS, EM, and EAB of 2% MgO increased significantly by 54, 48, and 13%, respectively. TS and EM of 4% ZnO increased, but EAB decreased significantly. TS and EM of the others did not change



**Figure 2.** The transmission of various PLA films in the range of 200–800 nm. (A) PLA films with MgO, (B) PLA films with NCC, (C) PLA films with ZnO, and (D) PLA films with MgO/ZnO.

significantly; hence, ZnO had a poor effect. PLA/MgO/ZnO showed a significant increase in TS, EM, and EAB at 1% by 21, 38, and 23%, respectively. The main mechanical properties of PLA films such as TS, EM, and EAB are reinforced (Wen et al., 2017).

The improvement of the mechanical properties of the PLA film by the nanofiller was mainly due to its large specific surface area, which promotes the stress transfer between the polymer molecular chain and the nanofiller (Arrieta et al., 2015). The smaller the size of the nanofiller, the larger the specific surface area. Therefore, the smaller the size of the nanofiller (MgO 50 nm and ZnO 100 nm) in the PLA film, the more obvious the improvement of the mechanical properties of the PLA film. The effect of nanofillers on the mechanical properties of PLA films was mainly determined by two aspects (Marra et al., 2016; Swaroop & Shukla, 2018). First, the addition of nanofillers to the polymer matrix provided a relatively high surface interaction between the filler and polymer chain, which helped the transfer of stress from the polymer chain to the nanomaterial, leading to the improvement of mechanical properties. Second, they tended to agglomerate because of the high surface energy of the nanomaterials, thus reducing the effective filler in the matrix, contrary to the first case. In addition, aggregated particles were beginning to behave like defects in the polymer networks; thus, adding more nanomaterials does not improve the mechanical properties of the films (Endres & Siebert-Raths, 2012; Shah et al., 2017).

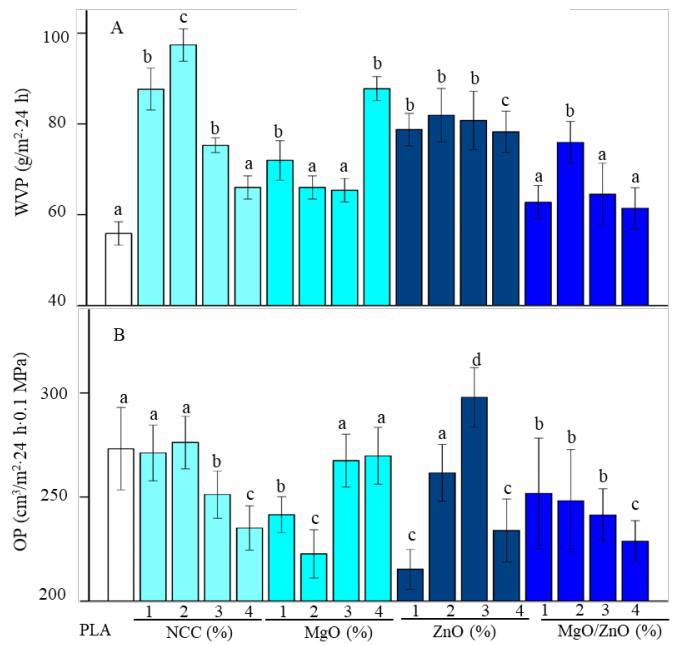


\*Different letters (a-c) indicate significant differences between groups.  
**Figure 3.** (A) EAB, (B) TS, and (C) EM of various PLA films\*.

### 3.3 Barrier properties of the nanomaterial-modified PLA films

Small molecular substances in the environment, such as water vapor and oxygen, can enter the internal environment through food packaging, thus leading to oxidation of foods and mass reproduction of microorganisms and causing food spoilage. Therefore, food packaging requires low permeability (Ciannamea et al., 2018). The barrier properties of packaging materials have a great impact on the shelf life of foods. Therefore, we measured the WVP and OP of the PLA films. The OP and WVP of all samples are shown in Figure 4.

The nano-modified PLA film has an oxygen transmission rate lower than that of the pure PLA film. The OP of PLA/NCC decreased with the increase of the addition and the 4% film decreased by 14%. The OP of PLA/MgO decreased first and then increased upon addition, and the OP of 2% film was the lowest (18% lower). The OP of the PLA/ZnO film increased first and then decreased and the OP of 1% film was the lowest (21% lower). The OP of PLA/MgO/ZnO film decreased upon addition and 4% addition produced the lowest OP value (21% lower). The above results show that the addition of materials can significantly reduce the OP of the PLA film, that the effect of MgO and ZnO was better ( $p < 0.05$ ), and that the OP of the PLA film can decrease by more than 20%, which may be because nanofillers hinder the diffusion of oxygen molecules (non-polar molecules) in the polymer, so it must bypass these nanofillers. This greatly extends the average diffusion path of oxygen molecules in the membrane and reduces the OP of the PLA film (Fabra et al., 2015; Galus & Kadzińska, 2016). In addition, the PLA film prepared in this study had OP higher than that in a similar study, which is likely due to the evaporation of the solvent during solvent casting, which resulted in a larger free volume and in turn promoted spreading of the gas molecules.



\*Different letters (a-c) indicate significant differences between groups.  
**Figure 4.** (A) WVP and (B) OP of various PLA films\*.

Compared with the WVP of the pure PLA film, the WVP of the nano-modified PLA film increased (Aydogdu et al., 2018). Among them, PLA/NCC film showed that WVP of the 2% film increased by 76%, the PLA/MgO film showed that the WVP of the 4% film increased by 58%, the PLA/ZnO film showed that the WVP of the 2% film increased by 47%, and the PLA/MgO/ZnO film showed that the WVP of the 2% film increased by 36%. According to the theory of molecular diffusion, nanofillers in polymer networks reduce the diffusion of small molecules but increase the diffusion of water molecules. This may be because the diffusion of water molecules (polar molecules) is not affected by the path effects (Aydogdu et al., 2018). In contrast, the interfacial effect of the nanofiller changed the absorption and dissolution characteristics of the free region in the polymer network, which in turn increased the permeability of the water molecule (Dashipour et al., 2015).

#### 4 CONCLUSION

In this study, the enhancement effects of ZnO, MgO, NCC, and ZnO/MgO mixture on PLA films were analyzed. The results show that nanomaterials were evenly distributed in the PLA film. PLA/ZnO and PLA/MgO/ZnO films had significant UV absorption effect. All of the nanomaterials could improve the mechanical properties of PLA film and the films with 2% MgO had the most remarkable enhancement effect ( $P < 0.05$ ); all of the nanomaterials decreased the OP and increased the WVP. The oxygen barrier property of the films with 1% ZnO or 2% MgO improved remarkably ( $P < 0.05$ ). Therefore, 2% MgO and ZnO enhanced the mechanical strength and oxygen barrier property of PLA film and endowed it with UV absorption capacity, which is conducive to promoting the application of PLA film in food packaging.

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