Investigation of the Influence of Enteric Polymers on the Moisture Barrier Properties of Opadry® 200

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Poster Reprint
AAPS 2013

Purpose

Opadry® 200, optimized performance coating, has previously been shown to be a versatile film coating that provides excellent moisture barrier and coating productivity.[1] In this study, the role of enteric polymers on the material properties and moisture barrier performance of polyvinyl alcohol (PVA) based Opadry 200 is investigated.

Methods

Materials

Fully formulated, immediate release film coating systems (Opadry 200, Colorcon Inc. USA) with different levels of enteric polymers (no, medium, and high), and polyethylene glycol (PEG) were studied. Two other fully formulated systems without PEG were also investigated (Opadry amb, aqueous moisture barrier film coating, and Opadry 200 with glyceryl monostearate (GMS) and a medium level of enteric polymer).

Film Preparation

A film coating dispersion was prepared by hydrating the formulated system in deionized water to 10% w/w solids. Films with thickness 100 ± 10 µm were prepared by spraying the dispersion onto a polyethylene terephthalate substrate. Film thicknesses were measured using a digital micrometer (IP54, Fowler Inc. MA, USA).

Glass Transition Temperature

A modulated DSC (Q200, TA Instruments, MD, USA) was used to determine the glass transition temperature of film samples. Film discs with a diameter of 4 mm were stored at 25°C and a relative humidity (RH) between 50 and 80% for 48 hours in open DSC pans. The pans were then sealed and thermograms immediately initiated between -40 and 160°C at a rate of 5°C/min and a modulation of 1.59°C/min.

Dynamic Vapor Sorption

Prior to testing, 10 mg film samples were pre-dried in an oven at 50°C and then placed in the dynamic vapor sorption analyzer (DVS Intrinsic, Surface Measurement Systems, PA, USA), where they were further dried at 0% RH until the mass variation was less than 0.001%/min.

Moisture sorption isotherms were determined at 25°C and 10–90% RH at 10% RH increments until the mass variation was less than 0.001%/min. The process was then reversed to obtain desorption isotherm data. The average moisture content from the sorption and desorption cycles was reported.

Water Vapor Transmission Rate (WVTR)

WVTRs were determined on sprayed films (thickness 100 ± 10 microns) using a VTI WPA-100 unit fitted with a 6.39 cm^2 test cell. Test films were first dried to an equilibrium dry state at 25°C with a dry N_2 purge gas flowing at a rate of 200 cm^3/min for 30 minutes. Moisture vapor was then introduced into the purge gas to develop relative humidity of 50, 65, 75 or 80% on one side of the test film. The moisture content on the opposite side of the test film was monitored, and the equilibrium rate of moisture transmission through the film was determined over a period of time. The test period was completed when the measured transmission rate deviated by less than 0.001 g water/day/m^2 for a period of 5 minutes.

Results

Glass Transition Temperature

The glass transition temperature (Tg) of PVA films as a function of relative humidity is listed in Table 1. The reduction in Tg at elevated relative humidity is indicative of the plasticizing effect moisture has on PVA. When a polymer is below its glass transition temperature it will be in a glassy, stiff state due to low molecular mobility, while above the glass transition temperature the polymer has greater molecular mobility. It would be anticipated that polymers in a glassy state will provide better moisture barrier performance than those in a rubbery state.[2,3]

Table 1. Glass transition temperature of pure PVA after storage at various relative humidity and 25°C.

RH (%)	Tg of PVA (°C)	Std Dev (°C)
0	69.9	0.0
50	24.4	0.1
65	8.3	0.0
80	-9.0	0.7

The glass transition temperatures of PVA and the fully formulated PVA-based systems following storage at 25°C and 65% RH are shown in Figure 1. It can be seen that the inclusion of PEG had a slight plasticizing effect on Opadry 200 no enteric as its Tg was lower than that of PVA alone. The inclusion of increasing amounts of enteric polymer into Opadry 200 tended to increase the polymer Tg.

PVA-based film coatings generally have excellent moisture barrier properties, and based on the Tg values, it would be anticipated that coatings with increased levels of enteric polymer would provide further reduced WVTR values; however, Figure 2 indicates that while the Tg for Opadry 200 high enteric was greater than that of Opadry 200 medium enteric, it actually had a slightly higher WVTR. Film formation may be affected by using different ratios of PVA to enteric polymer and explain the different moisture barrier performance at high enteric inclusion levels.

Figure 1. Glass transition temperature of PVA and formulated systems after storing at 65% RH and 25°C for 48 hours

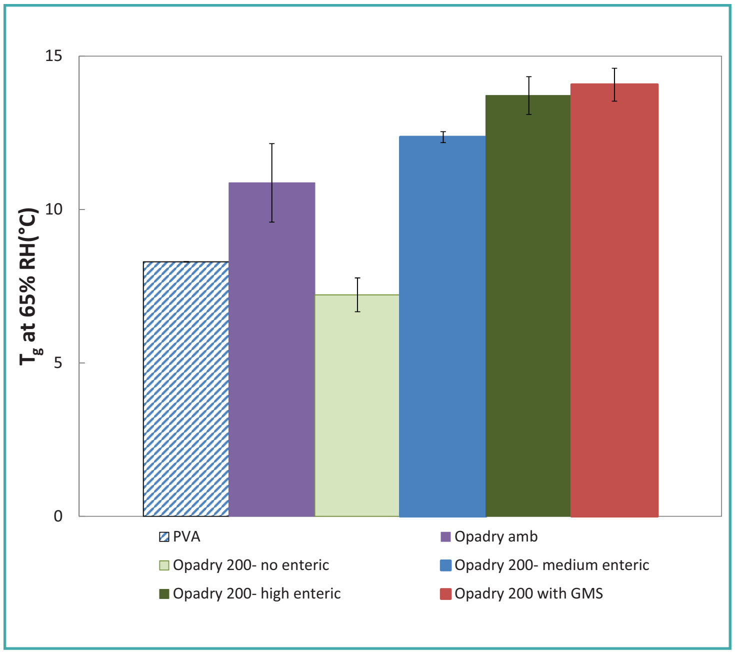
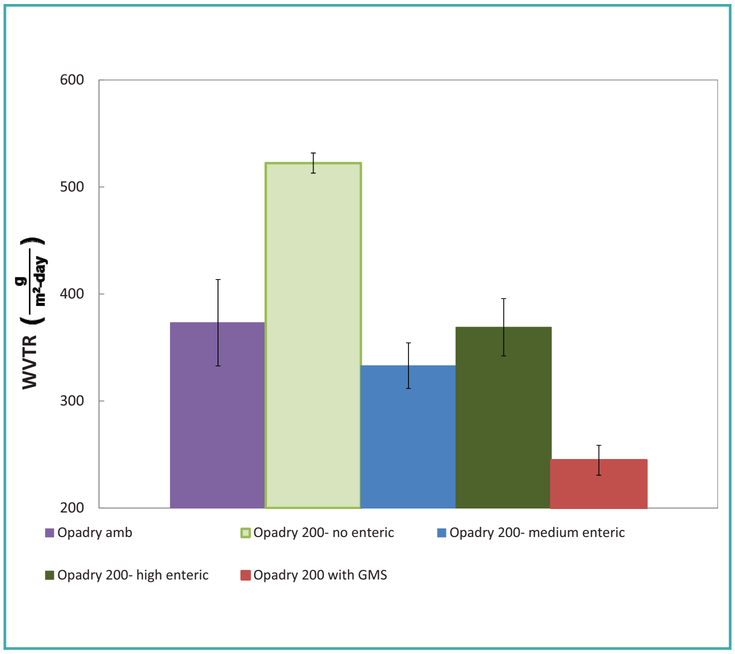


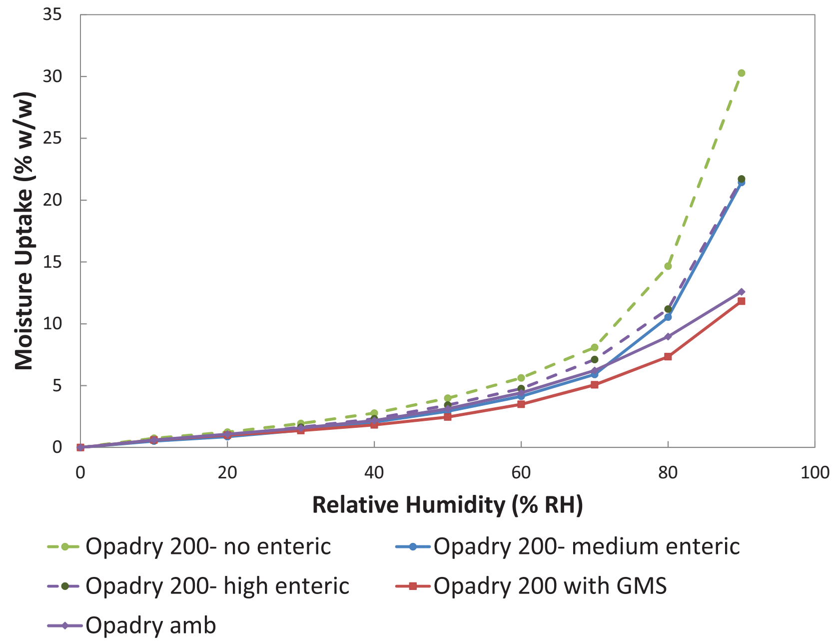
Figure 2. The influence of enteric polymers on the WVTR at 80% RH and 25°C for PVA-based film coating systems



Dynamic Vapor Sorption - Moisture Uptake

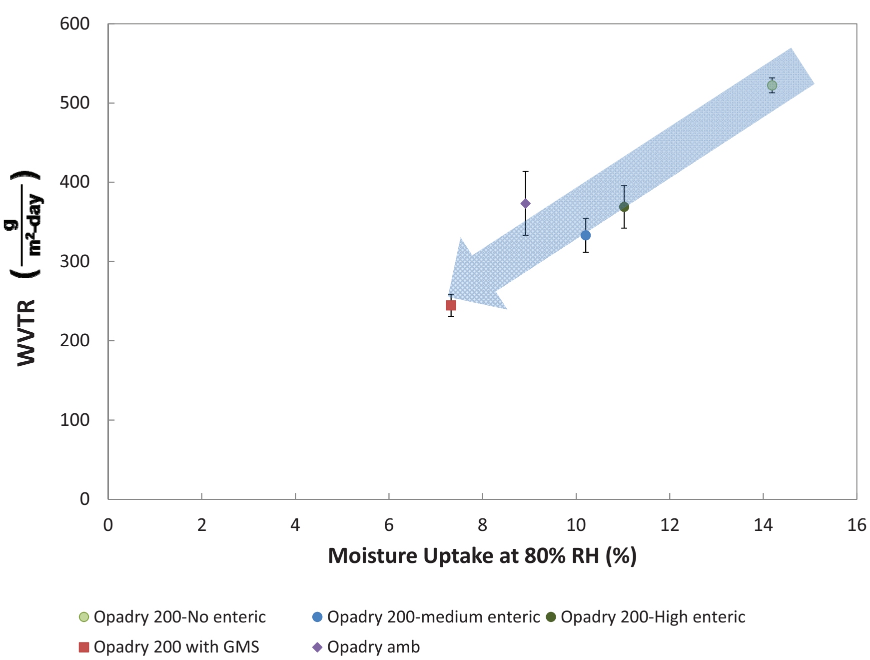
Moisture sorption isotherms of film coating samples are shown in Figure 3. Above 60% RH there was an apparent increase in moisture uptake for all systems. This behavior was more pronounced for the formulation that contained PEG and no enteric polymer, which had the highest moisture uptake levels. The two formulations without PEG (Opadry amb and Opadry 200 with GMS) exhibited lower moisture uptake levels even at high relative humidity, suggesting the hydrophilicity of PEG was an influencing factor. The combination of enteric polymer and glyceryl monostearate in Opadry 200 with GMS further reduced the moisture uptake compared to Opadry amb.

Figure 3. Moisture sorption isotherm (average over sorption and desorption cycle) for PVA-based films



The WVTR values and moisture uptake levels of the coating formulations at 25°C and 80% RH are compared in Figure 4. In general it can be seen that lower moisture uptake values are associated with lower WVTR values. This is likely due to the fact that higher moisture uptake levels will lead to larger moisture concentration gradients through the film thickness and accelerate moisture transmission rates.[4]

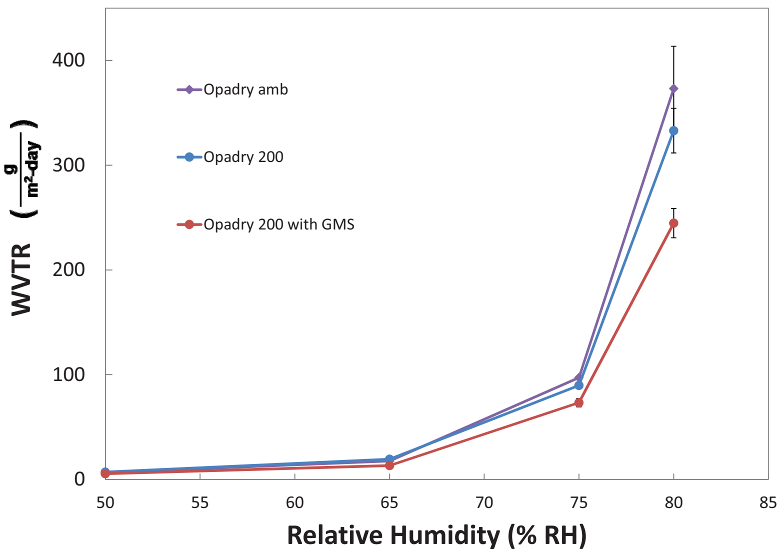
Figure 4. The influence of moisture uptake on WVTR values of PVA-based film coating systems



The Influence of % Relative Humidity on WVTR

The WVTR values of the fully formulated film coating systems are shown in Figure 5. All the film coating systems offer excellent moisture barrier performance below 65% RH, however some divergence in WVTR is observed at elevated relative humidity. The divergence can be associated with the plasticizing effect of moisture on the film, the hydrophilicity/ hydrophobicity of the coating formulation and the impact of enteric polymer increasing the Tg and glassy nature of the coating.

Figure 5. WVTR values of PVA based films at different relative humidity



Conclusions

Opadry 200 film coatings containing enteric polymers have been shown to exhibit superior moisture barrier performance. Formulations without PEG that include an enteric polymer had elevated glass transition temperatures and reduced WVTR values at elevated humidity. Formulations that exhibited lower moisture uptake values also had reduced WVTR.

References

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