



Development of a pectin-based oxygen barrier coating for food packaging applications

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ABSTRACT

Flexible food packaging films are challenging to recycle due to the complex multi-layer structures required for barrier properties for food protection, in a thin, transparent yet mechanically strong system. This work explores pectin as a bioderived oxygen barrier coating for polyethylene terephthalate (PET) films as a potential use case for meat packaging, targeting an oxygen transmission rate (OTR) below $1 \text{ cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$. Water-based coatings based on pectin were developed and deposited onto PET film. These coatings provided a viable oxygen barrier, reducing the OTR of $12 \mu\text{m}$ PET film from around $90 \text{ cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$ when uncoated to $\sim 2\text{--}4 \text{ cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$ when coated, a result comparable with ethylene vinyl alcohol (EVOH) currently in commercial use. Additionally, isopropyl alcohol (IPA) and graphene oxide (GO) were individually investigated as additives. GO addition was effective ($\text{OTR} \leq 0.7 \text{ cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$), but at the expense of reduced transparency. IPA addition resulted in the highest oxygen barrier with an OTR of $\leq 0.22 \text{ cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$. The tested pectin coatings were demonstrated to be water washable, allowing easy removal, leaving clean PET film for convenient recycling. This opens new opportunities for the use of PET as a viable thin film for packaging.

1. Introduction

Polymers are widely used across the food packaging industry, from flexible bags for fresh vegetables and dried goods such as rice and pasta, to rigid trays laminated with flexible films for meat. Packaging must be able to withstand the forces exerted on it through transportation and handling, extend the shelf life of the product to minimise food waste, display important product information, be transparent and prevent fogging. Plastic packaging is an indispensable resource for transporting food since its lightweight nature reduces transportation costs and its ability to extend shelf life reduces food waste. However, over 90 % of plastic produced relies on virgin fossil fuel feedstock (Ellen MacArthur Foundation and McKinsey and Company, 2016). In 2022, the UK produced 2.2 million tonnes of plastic packaging waste, with only 51.4 % of this recycled (Department for Environment, Food and Rural Affairs, 2024). This statistic alone does not reflect the clear disparity in outcomes between more recyclable rigid plastics and less recyclable flexible plastics. In a government research briefing, it was stated that in

2020/21, the UK recovered/recycled 47 % rigid pots/tubs and trays, but only 4 % of flexible films (Smith, 2024). Addressing the recycling of flexible films used in food packaging is therefore a crucial part of reducing overall reliance on virgin fossil fuel feedstock, but also preventing waste packaging being incinerated or sent to landfill.

Flexible films, such as those used in meat packaging trays, must possess several important characteristics such as mechanical strength, oxygen and water vapour barrier properties, seal strength, printability, antifogging tendency, transparency and thinness. The only practical way to achieve this currently is to make flexible composite films comprised of multiple polymers laminated together to create tailored functional properties (Fig. 1). These multilayer laminate flexible films are typically disposed of after a single use since effective recycling of multilayer film laminates requires the components to be separated into constituent polymers. This presents both technical and logistical challenges for collection and separation processes, which adds both cost and complexity to what is a low value and bulky waste stream and results in the vast majority of used flexible films being incinerated or sent to

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landfill. While bio-based flexible films offer a potential route to sustainability, due to their replenishable source and potential biodegradability, these are hampered by limitations in barrier and mechanical properties, especially where the high oxygen barrier properties are required e.g. protein packaging ($<1 \text{ cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$).

The composition of polymers can vary widely within a given composite, this adds to the cost and complexity of recycling operations (Kaiser et al., 2018). One method uses solvents to remove the adhesive “tie-layers” to delaminate the structure layer by layer (Cabrera et al., 2022). Correct identification of constituent polymers from the laminated structures after separation is vital to achieve a higher quality “clean” recycle, yet this identification imposes extra cost and complexity to an already low value waste stream.

Packaging films typically require a core structural polymer to impart mechanical strength to the film composite, with additional functional layers built on to this. Polyethylene terephthalate (PET) was identified in this study as a suitable carrier film since it is currently widely mechanically recycled and is often the chosen material for rigid packaging trays. These trays typically have a flexible thin film sealed to the tray to protect contents such as fresh meat. Although the tray is readily recyclable and forms the vast bulk of the post-consumer waste, the multi-component film which is heat sealed to it acts as a contamination which also reduces the efficacy of recycling in the tray itself. The use of mono-material PET lids would not provide sufficient oxygen barrier properties to the packaging and would reduce the shelf-life of the product. PET is regarded as a medium oxygen barrier among polymer films, with an oxygen transmission rate (OTR) of approximately $100 \text{ cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$ (H.-D. Huang et al., 2023; Joo et al., 2018), and a resulting oxygen permeability (OP) of $1200 \text{ cm}^3\cdot\mu\text{m}/\text{m}^2\cdot\text{day}\cdot\text{atm}$ (Hu et al., 2005; Joo et al., 2018; Wang et al., 2018). However, for high oxygen barrier food packaging, such as protein food packaging products, this is insufficient. In such applications, ethylene-vinyl-alcohol (EVOH) is commonly used in multilayer flexible film systems where the OTR of the multilayer film is less than $1 \text{ cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$ (Huang et al., 2023). However, the hydrophilic nature of EVOH renders it sensitive to moisture (Mokwena and Tang, 2012; Wu et al., 2021), which further dictates that it must be sandwiched between carrier films with interlayers of adhesives adding further complexity to the multi-layer system. Different EVOH grades with varying ethylene content are produced to improve sensitivity to moisture, where there is a trade-off with high ethylene content grade to reduce the water sensitivity (44 % ethylene content) EVOH having an OP of $23 \text{ cm}^3\cdot\mu\text{m}/\text{m}^2\cdot\text{day}\cdot\text{atm}$, and the lower ethylene content grade (32 %) having an OP of $5 \text{ cm}^3\cdot\mu\text{m}/\text{m}^2\cdot\text{day}\cdot\text{atm}$ at the expense of increased water sensitivity (Maes et al., 2018).

The use of often complex multi-material packaging films is dictated by the number of roles they must perform with limitations in terms of thickness and transparency. Mono-materials can be integrated into existing collection and recycling infrastructure, and this is reflected in their much higher recycling rate vs mixed plastic. Current mono-material films are unable to provide adequate oxygen and moisture barrier performance compared to multi-layer films. The ability to reduce the complexity of multi-layer films, while maintaining their superior

barrier properties, would aid their downstream recovery, reducing plastic packaging waste. Previous studies have coated PET with a bio-derived layer (chitosan/chitin nanowhiskers/cellulose nanocrystals) to improve the barrier properties which is removable in an alkaline wash (Ji et al., 2023; Yu et al., 2022). Yu et al. achieved an OP of $365 \text{ cm}^3\cdot\mu\text{m}/\text{m}^2\cdot\text{day}\cdot\text{atm}$, a 4-fold improvement compared to the uncoated PET substrate. Although the bio-derived layer reduced the oxygen permeability, the barrier is insufficient to replace existing barrier polymers such as EVOH in food applications. This work will therefore focus on improving the oxygen barrier properties of a PET film by developing a single polymer oxygen barrier coating (Fig. 1) suitable for meat applications, that can be readily washed from the film to leave a mono-material film that can be readily recycled.

In efforts to move towards a circular economy, the food packaging industry has explored alternative bio-sourced and biodegradable polymer products such as pectin, cellulose, chitosan, chitin, whey protein isolate, and starch (Fein et al., 2021a; Heidemann et al., 2019; Ji et al., 2023; Joo et al., 2018; Satam et al., 2018; Souza et al., 2022; Tyagi et al., 2021; Yu et al., 2022). A further factor when considering any materials that can reasonably be expected to be brought into contact with food, or to transfer their constituents to food, is that they must comply with the requirements of European Regulation (EC) No 1935/2004 (assimilated for Great Britain), so they do not endanger human health or bring about an unacceptable change in composition or organoleptic characteristics of the food. Pectin is therefore a promising potential material for this application since it is bio sourced, biodegradable, biocompatible, non-toxic, not derived from animal products, and low cost polysaccharide which is commercially extracted from food sources such as citrus peel, apple pomace and sugar beet pulp (Acar et al., 2024; de S. Medeiros et al., 2012; J. Huang et al., 2021; Jahangiri et al., 2024; Yang et al., 2024). Previous studies of pectin have indicated good oxygen barrier properties due to its tight network structure (Huang et al., 2021). Previous studies have tested 2wt % pectin (Siracusa et al., 2018; Souza et al., 2022; Ureña et al., 2023) cast in to a stand-alone film. Quoted oxygen permeabilities were 242 (Ureña et al., 2023), 17490 (Siracusa et al., 2018), and 23600 (Souza et al., 2022) $\text{cm}^3\cdot\mu\text{m}/\text{m}^2\cdot\text{day}\cdot\text{atm}$ for thicknesses varying from 50 to 110 μm . Despite possessing barrier properties, films cast from pectin would not have the appropriate mechanical properties to work as thin film packaging in themselves. However, despite the varied results, the biodegradable, non-toxic, water soluble, and film forming properties of pectin, make it a promising candidate polymer for further investigation, but as a coating to improve the barrier properties of a recyclable substrate. While coatings such as PVOH have been shown to offer potential for enhancing oxygen barrier properties of carrier substrates (Huang et al., 2012; Idris et al., 2021; Zhan et al., 2021), PVOH is not sustainable as it is fossil fuel derived.

In this work, pectin-based coatings were therefore developed to act as oxygen barriers for a PET carrier film. The solubility of pectin in water is advantageous as water-based coatings offer an environmental and food safety advantage, however, water-based coatings can be difficult to successfully coat on to hydrophobic polymers and are prone to dewetting. Alcohol was therefore investigated as a co-solvent and

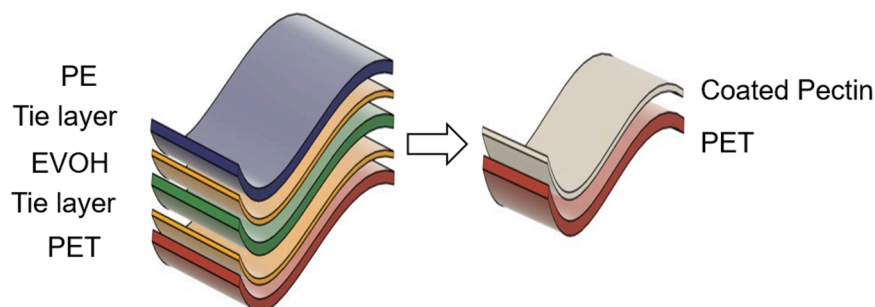


Fig. 1. An example multi-layer flexible film structure (left), where the thickness of each layer ranges between 5 to 30 μm , and proposed alternative structure (right).

wetting aid (Tåg et al., 2012) as unlike surfactants, it would not persist in the dry film.

Since there are conflicting demands of both high OTR and low layer thickness, additives may also play a role in providing acceptable performance while reducing the amount of barrier coating required. Nano materials have been investigated to improve the oxygen barrier performance of polymer films (Mishra et al., 2024). Graphene oxide (GO) has also been explored in literature as a potential additive to improve the oxygen barrier properties due to its high aspect ratio and water solubility (Alp et al., 2024; Han Lyn and Nur Hanani, 2022; Silva-Leyton et al., 2019). The addition of GO into a polymer matrix has been proposed to create a complex, tortuous pathway for oxygen diffusion, reducing the transmission rate (Han Lyn and Nur Hanani, 2022). However, the addition of GO has been reported to reduce the optical transmission through packaging films (Han Lyn and Nur Hanani, 2022). To the authors knowledge, the addition of graphene oxide to pectin and the effect on oxygen barrier properties has not yet been studied.

While pectin is derived from edible sources and is generally recognised as safe for food applications, graphene oxide (GO) is not yet approved for use in food packaging. However, its potential inclusion may be considered under existing regulatory frameworks that assess nanomaterials on a case-by-case basis, such as those outlined by the European Food Safety Authority (EFSA) or the UK Food Standards Agency (FSA). Further research in this area may prove useful in addressing current limitations in the design of sustainable food packaging and could also find applications in oxygen barrier technologies beyond the food sector.

This work investigates the formulation, deposition and barrier properties of water-based coatings incorporating pectin, testing also incorporation of GO as an additive. The viscosity of coatings was measured to consider suitability for industrial process range, deposited coatings were then characterised in terms of coating consistency, layer thickness, optical transparency with UV-Vis spectroscopy, surface chemistry via infrared spectroscopy, and finally oxygen transfer rate. Coatings were also assessed for their ability to be removed using hot water immersion.

2. Materials and methods

2.1. Materials

Polyethylene terephthalate (PET) films of 12 μm thickness were obtained from Klöckner Pentaplast (KP, Pontivy, France). Pectin, from citrus peel, was purchased from Merck (P9135). Graphene Oxide (GO) dispersion (1 %, aqueous, typical stated nominal size 5 μm in at least one lateral dimension, sheet depth <2nm (*Graphene Oxide Analysis*, n.d.)) was purchased from GOgraphene (Essex, UK). Isopropyl alcohol (IPA, IUPAC: Propan-2-ol) was purchased from Thermo Fisher Scientific. Methylene blue (MB) dye was purchased from Merck (M9140) and added in trace amounts to the coating solution for specific tests where it was required as a visual aid (for example in coating and washing tests) as otherwise the coating would be transparent. Deionised water with a resistivity greater than 1 M Ω -cm was used as the main solvent in the coatings. Potassium sorbate (PS) was purchased from Thermo Fisher Scientific and used to prevent microbial growth in the coatings. Preliminary experiments presented no visual microbial growth in the coating solution when 0.1wt % potassium sorbate was added. Without potassium sorbate, microbial growth was typically evident approximately seven days after formulation.

2.2. Preparation of pectin coating solutions

To make the coatings, deionised water was heated on a hot plate to 70°C. Once at temperature, pectin was slowly added and dispersed using an overhead mixer until it dissolved. The speed of the overhead mixer was increased from 400 to 800 rpm as polymer was added, and the

temperature of the solution was kept between 70–75°C. Once pectin had dissolved in solution, it was allowed to cool to laboratory temperature while still subject to overhead mixing. 0.1wt% potassium sorbate and 0.01wt% MB dye (if required) was then added.

To incorporate IPA into the coating, 10 wt % was added gradually to the prepared and cooled pectin solution under overhead stirring. For the incorporation of GO dispersion into the coating, the GO dispersion underwent sonication with a Sonopuls ultrasonic homogenizer probe (Bandelin electronic GmbH & Co, KG) for 30 minutes. The dispersion was added to deionised water prior to adding pectin. The composition of each coating is listed in Table 1.

2.3. Deposition of pectin coatings

The application of the pectin coating onto the surface of the PET was carried out via bar coating using an RK control coater (K101 control coater, RK print coat instruments). The PET substrates were wiped with an IPA soaked, lint-free cloth to remove any potential surface contaminants. Two wire wound bars of differing wire thicknesses (L=lower, H=higher) were used to coat different coating thicknesses: a 0.3 mm diameter wire (indicative wet film thickness 24 μm ; P-(L)) and a 0.64 mm diameter wire (indicative wet film thickness 50 μm ; P-(H)). The approximate film thickness after drying was 2 and 4 μm , respectively. The different thicknesses allowed an evaluation of potential trade-offs between the use of raw material and oxygen barrier properties. After deposition, the coated substrates were dried in a box oven at 90°C for 10 minutes. Table 2 details the combinations of wet film thickness and coating formulations used in the study. For brevity a shorthand sample identification is also listed.

2.4. Evaluation of washability of coating from substrate

As part of the recycling process, any potential oxygen barrier coating would require removing from the base recyclable substrate. To evaluate washability, coated samples (sized 5 × 5cm) were immersed in 250ml of hot water (~70–80°C) for 10 minutes, an effective temperature to remove the coating and replicable to recycling wash procedures (Lase et al., 2022). Washed samples were inspected visually, with the absence of colour from the methylene blue dye used to indicate the removal of coating. Infrared spectroscopy was used to determine the presence or absence of coating in the non-dyed state.

2.5. Characterisation techniques

2.5.1. Rheometry of coating dispersions

The rheology of the coatings was characterised using a stress-controlled rheometer (Kinexus Pro Rheometer, Netzsch) with a 50 mm stainless steel 1° cone and flat plate geometry at a constant temperature of 25±0.1°C. The shear viscosity was measured at shear rates between 0.1 and 10 s⁻¹. Small amplitude oscillatory shear (SAOS) measurements were taken to indicate the viscoelastic properties of the fluids, with a strain amplitude sweep used to identify the viscoelastic properties of the fluids at 1 Hz, with values for the phase angle taken from within the linear viscoelastic region (LVR) region (Claypole et al., 2020) at a constant frequency of 1 Hz.

Table 1
Coatings developed in the study.

Composition name	Coating composition (wt%)			
	Pectin	Deionised Water	GO	IPA
Pectin	8	92	0	0
Pectin-IPA	8	82	0	10
Pectin-GO	8	92	0.1	0

Table 2
Sample identification and composition.

Sample ID	Name	Coating composition	Nominal Wet film coating thickness (μm)
PET	Control	No coating	0
P-(L)	Pectin 24 μm nominal	Pectin	24
P-(H)	Pectin 50 μm nominal	Pectin	50
P-IPA	Pectin with IPA	Pectin-IPA	50
P-GO	Pectin with GO	Pectin-GO	50

2.5.2. Visual inspection of coated samples

The consistency of coating deposition was evaluated visually since an inconsistent, or poorly deposited coating layer could result in poor oxygen barrier properties. To assess the coating consistency, coated substrates were imaged using a desktop scanner (Epson Perfection V700) at 600dpi and 24-bit colour. After the coated substrates underwent the wash cycle, the samples were re-scanned to visualise the efficacy of the removal process.

2.5.3. Measurement of dry coating thickness

The thickness of the coated substrate was measured to calculate the oxygen permeability (from the OTR) and to normalise this with respect to the varying film thickness. An induction gauge (Laserliner®) was used to perform the thickness measurement of the samples with an accuracy $\pm 3\% + 1\mu\text{m}$. Three samples of each coating variation, and uncoated substrate, were measured with 5 measurements taken per sample ($n=15$).

2.5.4. UV-Vis and IR spectroscopy of coating layers

To determine the effect of the coating on light transmission through the coated films, UV-visible spectra of the PET film and coated films were recorded using UV-Vis-NIR spectrophotometer (Lambda 750, Perkin Elmer) in the range of 200 – 800 nm. Three samples of each coating variation were measured with 5 measurements taken per sample ($n=15$).

Fourier transform infrared spectroscopy (FTIR) in attenuated total reflectance (ATR) mode was used to detect any potential changes to the functional groups on the surface from the addition of IPA and GO, and to confirm the presence or otherwise of the transparent coatings. Measurements were taken using FTIR spectrometer (Frontier, Perkin Elmer). FTIR spectra were collected in the range of 4000 to 650cm^{-1} at a resolution of 0.4cm^{-1} and represent an average of 16 scans. The equipment was also used to assess the removal of pectin coatings from the surface of PET after washing.

2.5.5. Oxygen barrier properties of coated films

The oxygen transmission rate (OTR) was determined using an OTR permeation analyser OX-TRAN® 2/12(R) (MOCON) according to methods prescribed in ASTM F2622 (ASTM International, 2020) at the Biocomposites Centre, Bangor University. Testing was carried out at 23°C and 0% RH, with an exposed test area of 50cm^2 . Measurements were carried out at atmospheric pressure. The equipment is suitable for detecting OTR levels with a quoted lower limit of detection of $0.05\text{cm}^3/\text{m}^2\cdot\text{day}$, below which any results are shown as <0.05 (not detectable). For this study, OTR lower than $1\text{cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$ was considered acceptable for protein food packaging applications (Huang et al., 2023; Ureña et al., 2023). The OTR was expressed as $\text{cm}^3/\text{m}^2\cdot\text{day}$. The OTR was then normalised to the pressure difference, which in this study is 1 atm, as displayed in Eq. (1), referred to as permeance (Perm) expressed as $\text{cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$ (Ge et al., 2023). The oxygen permeance of the individual pectin-based coated layers was calculated according to equation 2, to calculate the permeability coefficient of the individual pectin-based layers. The oxygen permeability coefficient is calculated by multiplying the permeance with the average thickness (L) as

described in Eq. (3). The oxygen permeability coefficient is expressed as $\text{cm}^3\cdot\mu\text{m}/\text{m}^2\cdot\text{day}\cdot\text{atm}$.

$$\text{Permeance} = \frac{\text{OTR}}{\Delta p} \quad (1)$$

$$\frac{1}{\text{Permeance (coated film)}} = \frac{1}{\text{Permeance (substrate)}} + \frac{1}{\text{Permeance (coating)}} \quad (2)$$

$$\text{Oxygen permeability coefficient} = \text{Permeance} \times L \quad (3)$$

3. Results

3.1. Rheology of coating dispersions

The viscosity of the coatings at shear rates between 0.1 and 10 s^{-1} is shown in Fig. 2a. Phase angle data can be found in the supplementary documentation. Changes to the coating formulations had a large effect on the viscosity and flow behaviour of the coatings (Fig. 2a). The 8wt % pectin solution in water only showed near-Newtonian behaviour with a shear viscosity of $\sim 11\text{ Pa}\cdot\text{s}$ at 1 s^{-1} and the addition of $0.1\text{wt}\%$ of GO increased the viscosity of the coating by a factor of three to $\sim 33\text{ Pa}\cdot\text{s}$ at 1 s^{-1} . The use of 10% IPA co-solvent had a more substantial effect on the viscosity and the flow behaviour of the coatings, with an increase in shear thinning behaviour over the shear rates measured and an increase in viscosity to over $100\text{ Pa}\cdot\text{s}$ at a shear rate of 1 s^{-1} .

In terms of visco-elastic characterisation (see supplementary information Fig. s1.), 8wt % pectin in water gave a phase angle of 72° at 1 Hz , suggesting predominantly liquid-like behaviour. The addition of $0.1\text{wt}\%$ GO to the coating decreased the phase angle to 64° , indicating a transition to more elastic like, though still predominantly viscous behaviour, likely due to increased elastic interactions between GO particles and between GO and pectin. Use of IPA co-solvent significantly reduced the phase angle to 50° , with elastic behaviour becoming more significant.

3.2. Coating consistency and thickness

Scanned images of coated substrates are shown in Fig. 2c. Images are presented for coating with and without methylene blue as a visualisation aid. All four pectin-based coatings wetted out onto the surface forming a homogeneous film over the substrate as evidenced by the consistent blue colour from the dye.

Thickness data for the PET film with and without coating is shown in Fig. 2b. The average thickness of the non-coated film was $13.9 \pm 1\mu\text{m}$ ($n=15$). The average thickness of the films coated with the 0.3 mm wire diameter bar was $15.31 \pm 0.86\mu\text{m}$ ($n=15$), and $17.65\mu\text{m} \pm 1.47$ for the 0.63 mm wire diameter bar. There was variability in the measured thickness of PET, and therefore similar variability existed in the coated substrate measurements. On subtracting the average thickness of the substrate, the overall dry coating thickness is estimated around $1.4\mu\text{m}$ and $3.7\mu\text{m}$ for 0.3 and 0.6 mm bars respectively. There was no significant difference in thicknesses between coatings using pectin in water alone (P-(H)), in water with graphene oxide (P-GO), or in water and isopropyl alcohol (P-IPA) when the 0.63 mm wire diameter bar was used.

3.3. UV-Visible spectra of coated films

UV-Vis spectra are compared for coated and uncoated PET film in Fig. 2d, while light transmission at 400 nm is tabulated in the supplementary documentation (Table s1). Unless graphene oxide was used in the formulation there was no apparent reduction in light transmission in the 400 to 800 nm (visible) part of the spectrum when coatings were applied to the PET. The increase in thickness between coatings produced

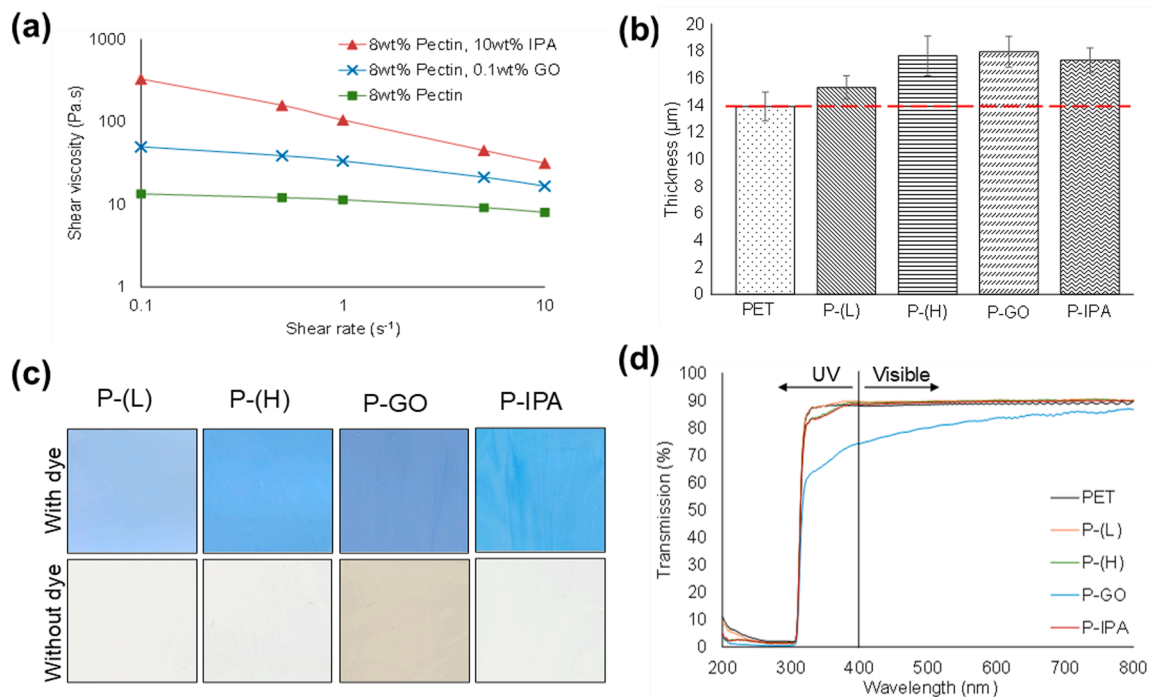


Fig. 2. (a) The equilibrium shear viscosity of 8wt% pectin suspensions at shear rates between 0.1 and 10 s⁻¹. The shear viscosity data was taken at 3s intervals over 30s at each shear rate with an average of each data point displayed. (b) Comparison of thickness measurements between samples. Red line represents the average thickness of PET, to assess the relative thickness of the different coatings. (c) Scanned images of pectin coated substrates comparing coating consistency with dye (sample size 5 × 5cm) (top) and transparency of coatings (sample size 2.5 × 2.5cm) (bottom). (d) UV-Vis spectra for each coated sample.

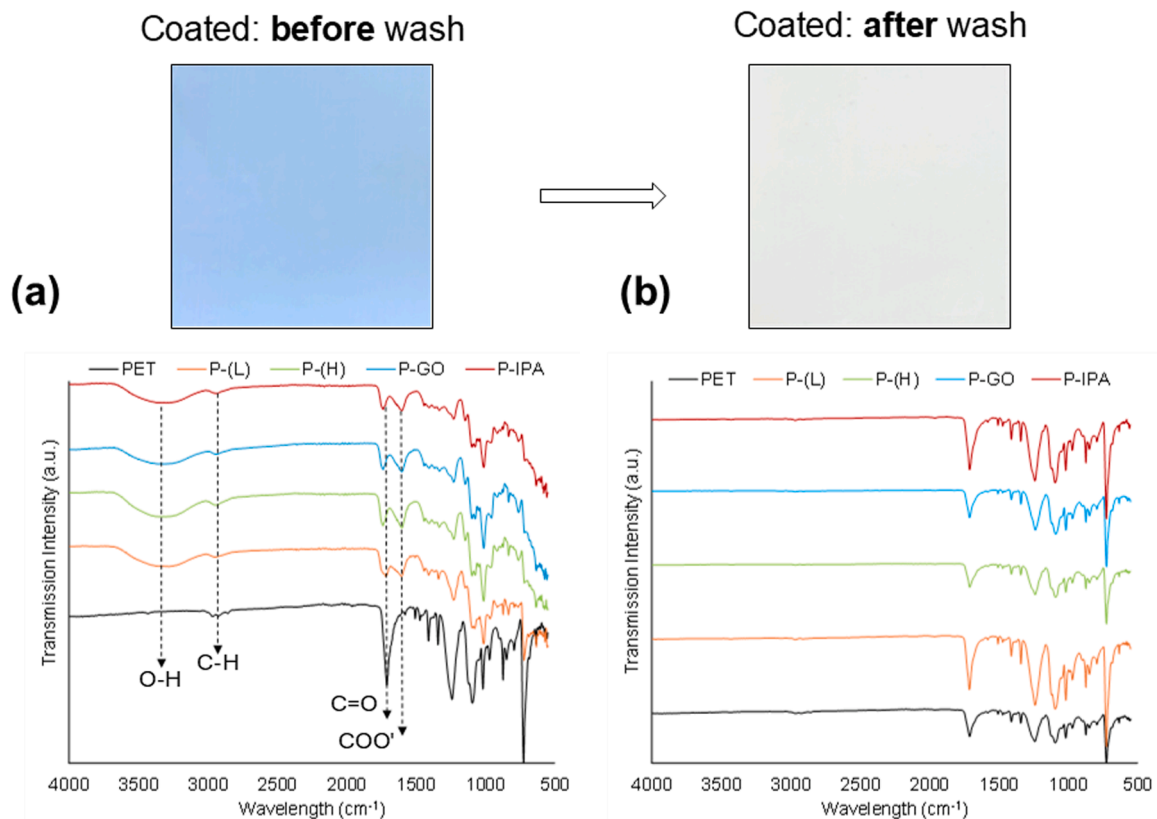


Fig. 3. Scanned images (top) size 5 × 5cm show appearance of dyed coating before (a) and after washing (b). FTIR spectra (bottom) of PET and pectin-based coated samples on PET film (a) before washing, and (b) after washing at ~70-80°C (where all spectra indicate no coating remaining on PET film).

from the 0.3 mm wire diameter bar and the 0.63 mm wire diameter bar led to a slight decrease in transmission between 320 and 380 nm, however at 400 nm there was little variation observed between all three pectin coating samples. This suggests that the addition of a pectin-based coating layer should not have any adverse effect of transmission through the film. Incorporation of graphene oxide into the coating gave it a grey hue which had a significant effect on light transmission, with reduced light transmission throughout the visible range, and at 400 nm, the light transmittance was reduced from 87.7 % for the non-coated PET, to 73.5 % for the pectin-graphene oxide coating (a reduction of 14.2 %). This would noticeably affect the appearance of the product.

3.4. Infrared spectra of coated films

Infrared spectra are compared for uncoated PET film and coated and dried films in Fig. 3a. The PET substrate has a strong peak at 1700 cm^{-1} representing the C=O functional group. Pectin coated substrates showed a broad band between 3600 and 3000 cm^{-1} indicative of the hydroxyl bond (-OH), a key identifier of pectin (Bhatia et al., 2024; Karim et al., 2022; Santos et al., 2020; Wang et al., 2014) which is not present in PET which lacks a hydroxyl group in its structure. The small peak between 3000 and 2900 cm^{-1} corresponds to the absorption of C-H bonds and is therefore found in both coated and uncoated PET (Bhatia et al., 2024; Karim et al., 2022; Santos et al., 2020). The two peaks at around 1770 and 1520 cm^{-1} are associated with ester carbonyl groups and carboxylate stretching (Bhatia et al., 2024; Karim et al., 2022; Santos et al., 2020; Wang et al., 2014), with the former more strongly evident in PET and the latter observed in the coatings. There was no apparent difference in infrared absorption between the different thicknesses of pectin, or when using isopropyl alcohol as a co-solvent. The functional groups in graphene oxide are similar to pectin (Bera et al., 2018), and therefore the presence of GO could not be distinguished in the coating. It is envisaged that this new approach opens new opportunities for the use of PET as a viable thin film for food packaging.

3.5. Oxygen barrier properties of coated and uncoated PET films

The OTR data are provided in terms of cm^3 of oxygen per m^2 per day in Table 3, three samples are reported per coating type. The term n.d. (non-detectable) signifies that the OTR was below the $0.05\text{ cm}^3/\text{m}^2\cdot\text{day}$ detection limit of the equipment. The uncoated $12\text{ }\mu\text{m}$ PET film gave an OTR of $\sim 92\text{ cm}^3/\text{m}^2\cdot\text{day}$. Using the measured film thickness of $13.9\text{ }\mu\text{m}$ gives a resulting oxygen permeability (OP) of $1293\text{ cm}^3\cdot\mu\text{m}/\text{m}^2\cdot\text{day}\cdot\text{atm}$ which is in line with previously published data for PET (Hu et al., 2005; Joo et al., 2018; Wang et al., 2018). All the pectin-based coatings substantially improved the oxygen barrier properties and measured OTRs were below $4\text{ cm}^3/\text{m}^2\cdot\text{day}$ in all cases, an improvement of over 20 times. The thinner pectin only coating ($1.4\text{ }\mu\text{m}$ dry film) gave an average oxygen barrier of $3.44\text{ cm}^3/\text{m}^2\cdot\text{day}$, which is considered a good oxygen barrier (H.-D. Huang et al., 2023; Ureña et al., 2023). OTR was improved

by using a thicker pectin layer ($3.7\text{ }\mu\text{m}$ dry film) and improved further still with the addition of graphene oxide or isopropyl alcohol which reducing the oxygen transmission rates to below $1\text{ cm}^3/\text{m}^2\cdot\text{day}$ or below detectable limits in some samples. These results are indicative of over a hundredfold improvement in oxygen barrier capabilities with moderately thin coating layers.

3.6. Assessment of coating removal

Infrared spectra of coated samples before and after washing are presented in Fig. 3. After washing, all four coatings were removed from the substrate, as evidenced by the lack of hydroxyl groups (3600 and 3000 cm^{-1} wavenumber) evident in the infrared spectra and lack of colour in the dyed coated samples (see also supplementary material Fig. S2.). This indicates that the coatings can be readily cleaned from the PET to allow for recycling.

4. Discussion

4.1. Oxygen barrier performance

A series of water-based pectin coatings were developed and applied to PET and were found to give substantial improvements to the oxygen barrier performance. A thinner pectin only layer (dry film thickness $1.4\text{ }\mu\text{m}$) reduced the oxygen transmission rate (OTR) of PET from $92\text{ cm}^3/\text{m}^2\cdot\text{day}$ to below $4\text{ cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$ for coated PET, while a thicker layer gave a further reduction in OTR to $2.3\text{ cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$ or below. These levels can be considered indicative of good but not high oxygen barriers (Ureña et al., 2023). However, a further improvement was provided through the incorporation of graphene oxide (GO) or use of isopropyl alcohol (IPA) as a co-solvent in the predominantly water-based dispersion. These modifications gave OTR values lower than $1\text{ cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$ which is indicative of high oxygen barrier performance necessary for replacement of existing barrier materials such as EVOH (H.-D. Huang et al., 2023; Ureña et al., 2023). Pectin-based coatings achieved similar barrier properties to equivalent EVOH, while pectin also offers the key attributes of being bio-derived, sustainable and bio-degradable. Previous studies of pectin as an oxygen barrier have only focussed on use as a stand-alone cast film (Siracusa et al., 2018; Souza et al., 2022; Ureña et al., 2023) and the use of a carrier film will provide the mechanical strength that is required for food packaging applications. The oxygen permeability for the individual pectin layers were calculated using the average film thickness for each sample. Therefore, the permeabilities are indicative comparisons against EVOH as a barrier. Although pectin without additives provides a similar barrier against oxygen permeability to EVOH, both pectin with IPA and GO were found to be consistently more impermeable to oxygen than pectin as a stand-alone coating. The oxygen permeability of both pectin with IPA and pectin with GO were below the cited permeability of EVOH of $5\text{ cm}^3\cdot\mu\text{m}/\text{m}^2\cdot\text{day}\cdot\text{atm}$ (Maes et al., 2018).

Table 3

Oxygen transmission rate and permeability coefficient results of coated samples, OTR results for each sample displayed. Test conditions 23°C , 0% RH.

Sample #	OTR $\text{cm}^3/\text{m}^2\cdot\text{day}$			Pectin coating permeance** ($\text{cm}^3/\text{m}^2\cdot\text{day}\cdot\text{atm}$)	Average coating thickness (μm)	Oxygen permeability coefficient ($\text{cm}^3\cdot\mu\text{m}/\text{m}^2\cdot\text{day}\cdot\text{atm}$)
	Sample 1	Sample 2	Sample 3			
Control (PET only)	92	92.98	91.44	n/a	n/a	n/a
Pectin $24\text{ }\mu\text{m}$ nominal (P-(L))	3.22	3.32	3.79	3.95	1.41	5.57
Pectin $50\text{ }\mu\text{m}$ nominal (P-(H))	2.31	2.05	<0.05*	2.37	3.75	8.88
Pectin with GO (P-GO)	0.11	<0.05*	0.69	0.70	3.43	2.82
Pectin with IPA (P-IPA)	<0.05*	<0.05*	0.22	0.22	4.06	0.76

* Less than $0.05\text{ cm}^3/\text{m}^2\cdot\text{day}$, below the detection limit of the equipment

** Calculated using the highest measured OTR for each sample

4.2. The effect of additives on performance

GO as a nano-particle is proposed to increase the tortuous pathway for oxygen to permeate through the film (Han Lyn and Nur Hanani, 2022). While this is indicative of high barrier performance required by the market, a disadvantage of adding GO is the reduction in transparency of the film and grey hue provided by the material. Research from consumer studies suggests packaging design, such as transparent windows, affect the consumer perception of the packaged product. Therefore, tinted/reduced transparency may affect consumer attitudes to the products packaged within (Simmonds et al., 2018). Despite this limitation, GO can improve barrier properties with only a very small addition, and this could be exploited where transparency is not a critical property.

A possible mechanism for the increased barrier performance when using isopropyl alcohol as a co-solvent may be due to an increasing in aggregation of pectin chains within the structure, making a stronger network (Jiang et al., 2020, 2021). This change in conformation was also evidenced by the changes to the viscoelastic properties of the coating solution likely due to the alcohol decreasing the zeta potential of the pectin chains, causing chain aggregation and bundling, leading to a resultant increase in the shear viscosity of the coating (Jiang et al., 2020, 2021; Liu et al., 2018). The variation in viscosity and more gel-like behaviour would have to be considered when transferring this technology to industrial coating lines (Han et al., 2009; Kipphan, 2001; Morgan et al., 2018).

4.3. Recyclability

The primary purpose of this study was to provide an effective oxygen barrier that could be more readily separated from the carrier film to permit recycling of a product that is usually sent to landfill or incinerated. Immersion at ~ 70 – 80°C was sufficient to remove any trace of the pectin-based coatings. This temperature is typical for industrial washing of used plastics such as PET but can be varied depending on the temperature response of the polymer. Following successful separation from the substrate, the PET film can be mechanically recycled without contaminants on the surface. The coating should be applicable to a range of carrier substrates (e.g. polyethylene, polypropylene) which can offer complementary properties such as acting as a water barrier, but the coating suitability would require verification in terms of aspects such as wetting. The approach of coating recyclable substrates with bio-based polymers enables customisable properties for flexible films without compromising the functional properties required of flexible food packaging films. A hot wash step can be readily introduced into a conventional recycling process.

4.4. Limitations

Although pectin presents a bio-based, biodegradable alternative to EVOH and other high oxygen barrier polymers, the long-term performance and scale up challenges are unknown and are to be investigated. Due to the bio-based, water soluble nature of pectin it is likely to be sensitive to changes in relative humidity. As with synthetic polymers such as EVOH and PVOH, contact with water is likely to reduce the oxygen barrier performance of a pectin based coating. Therefore, although a single deposited coating may not currently be suitable for all flexible packaging applications, as an intermediate measure, pectin could replace synthetic polymers of equivalent high oxygen barrier performance in a multilayer structure. Investigations to improve the water resistance of bio-degradable polymers, such as PVOH, are ongoing and additives such as GO, citric acid, boric acid, and glutaraldehyde have been shown to reduce swelling and dissolving in contact with water (Lai et al., 2015; Park et al., 2022; J. Wang et al., 2011; Z. Wu et al., 2017). However, the addition of additives can raise additional challenges, in particular for food contact applications. For example, the use

of GO in food packaging applications may be limited due to the reduction in transparency, but more importantly from a food safety perspective, the current risks of any migration into the food products are unknown. In terms of barrier performance, GO offered a benefit over pectin alone, but this might not justify the potential risk of integrating GO into any layer that could come in to contact with food, especially since formulating the pectin coating with IPA gave even better performance. Studies have reported that the biocompatibility and toxicity of nanomaterials such as GO is associated with their shape, size, and dosage. (Han Lyn and Nur Hanani, 2022; Rovera et al., 2020) The addition of GO in a biodegradable polymer may also pose challenges in terms of the prevention of the release of nanoparticles into the environment, and subsequent impact on aquatic life (Hashim et al., 2022). However, due to the potential improvement in barrier performance of bio-based coating that GO can provide, this system could be transferred to other applications e.g. electronics. Previous work has also tested whether there is any negative effect on the barrier properties of biobased polymers after folding (Fein et al., 2021b). This study reported there was no deterioration of the water vapour barrier properties after folding, though this would depend on the combination of carrier film and coating. While the application as a film lid is unlikely to result in excessive bending, any application that may require bending, folding, or use in thermoforming, for example, would require additional testing. The introduction of coating into an existing production line will require consideration, and although the coated layer is relatively thin, the introduction of a water-based coating will require drying processes and associated energy expenditure.

4.5. Future work

This study presents an industrially scalable solution, where the coating can be deposited using high speed roll to roll coating lines. In the longer term the precise coating composition may require further investigation or modification according to the selected coating process and required barrier performance. Further testing is required to verify performance in humid environments. The incorporation of additives could further improve aspects such as processability, coating performance, film properties, plus resilience to moisture and humidity. Although potassium sorbate was added as a preservative to prevent microbial growth and spoilage, the interaction of food borne microbes within the layer should be investigated further. However, the key benefits of this approach lie in the use of a sustainable, bio-derived, biodegradable source material and the ability to remove with simple washing.

5. Conclusions

Flexible packaging films are challenging to recycle due to the complex multi-layer structures required for food protection, in a thin, transparent yet mechanically strong system. This work demonstrated an oxygen barrier coating solution based on pectin, a bio-derived and degradable polymer, deposited onto polyethylene terephthalate (PET) carrier film, with comparable oxygen barrier properties to existing multilayer laminate structures. The results highlighted the excellent oxygen barrier performance achieved with pectin at coating depositions less than $4\ \mu\text{m}$ thick. These coatings provided a viable oxygen barrier, reducing the oxygen transfer rate of $12\ \mu\text{m}$ PET film from around $90\ \text{cm}^3/\text{m}^2.\text{day.atm}$ when uncoated to around $2\text{--}4\ \text{cm}^3/\text{m}^2.\text{day.atm}$ with a pectin coating. This work shows that the addition of IPA to pectin increases the oxygen barrier properties to levels comparable to ethylene vinyl alcohol (EVOH) with oxygen permeability less than $0.76\ \text{cm}^3.\mu\text{m}/\text{m}^2.\text{day.atm}$, showing required oxygen barrier performances for food packaging applications can be achieved with a bio-sourced, biodegradable polymer. Coatings were water washable at ~ 70 – 80°C , leaving clean PET film for convenient recycling. This novel approach to achieving a recyclable flexible film packaging system is a step towards a circular

economy for food packaging. Although its current application has not been tested in humid environments, pectin coated substrates may be suited for environments less sensitive to humidity, such as baked goods, and a green alternative to synthetic high barrier polymers such as EVOH as a replacement barrier layer. Future research to improve the water sensitivity of bio-based polymers, for instance with the use of non-toxic crosslinkers could address these limitations and expand this technology into applications such as protein packaging.

Ethical statement

The authors declare that the work in this paper does not involve studies involving humans or animals, no ethical approvals were required as part of this research.

CRediT authorship contribution statement

Ffyon Moody: Writing – original draft, Methodology, Investigation, Conceptualization. **Andrew Claypole:** Writing – review & editing, Methodology, Conceptualization. **Jenny Woods:** Writing – review & editing, Investigation. **Ben Clifford:** Methodology. **Qiuyun Liu:** Supervision. **Craig Hardwick:** Supervision, Resources, Project administration. **Chris Phillips:** Writing – review & editing, Validation, Supervision, Project administration, Methodology. **Davide Deganello:** Writing – review & editing, Validation, Supervision, Project administration, Methodology, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

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Data availability

Data will be made available on request.

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