



Work Package 3

Deliverable D3.5-Monomaterial Packaging Design

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Project summary

At STOPP, we're pioneering a transformative initiative to revolutionize the way we approach food packaging by working with all the stakeholders in the value chain to promote the "5 Rs": Refuse, Reduce, Redesign, Reuse, and Recycle

Our main aim is to drastically reduce the environmental impact caused by plastic waste in food packaging, aligning closely with the EU's Packaging and Packaging Waste Directive. Our collaborative efforts encompass every facet of the food packaging value chain.

We're set on creating circular strategies that not only appeal to plastic production and processing but also drive awareness through a multi-actor network. Our strategic actions include analysing plastic waste impact, monitoring current usage, designing sustainable business models, boosting recycling efforts, and understanding consumer attitudes through an in-depth study.

Executive summary

This report is prepared in alignment with EU STOPP deliverable D3.5, Monomaterial packaging development to facilitate recyclability of flexible plastic food packaging. Flexible films have a major share in food packaging industry for safety and shelf life. The EU Packaging and Packaging Waste Regulation (PPWR) mandates all packaging to be recyclable by 2030. The state-of-the-art multifunctional films such as Polyethylene Terephthalate – Polyethylene – Polypropylene (PET-PE, PET-PP) impose various sorting and recycling challenges. PP offers good physical and aesthetic properties, outstanding barrier for water vapour but limited Oxygen barrier which is critical for food shelf life. The PPWR and supporting design for recycling (DfR) standard EN 18120-7 (CEN-2026) enforce all flexible PP-PE as monomaterial without Oxygen barrier PET layer in the material composition. The RecyClass protocol on Recyclability Evaluation defines PP monomaterial films to be 90% composed of PP with maximum thickness of 250 μm . The EN 18120-7 guidelines prohibit use of barrier coating such as aluminium foil. Additives which do not impede recycling and reuse can be compounded with PP at weight percentage less than 7.5. These include for instance aluminium and silicon oxides AlOx , SiOx , and Ethylene Vinyl Alcohol (EVOH).

The research objective is to improve the Oxygen barrier level of monomaterial PP films based on film thickness enhancement, modulating cast film and biaxial stretching parameter, and additives. The methodology is based on full factorial design and Pearson correlation to systematically investigate distinct and synergetic sensitivity of BOPP processing parameters to barrier properties.

The optimum BOPP barrier performance is realised in 40-50 μm thickness range with further 50% increase 50% at 100-120 μm range beyond which there is no major improvement. The BOPP crystallinity and barrier properties is highly sensitive and dependant on synergetic effect of stretch ratio and processing history controlled by chill roll temperature. Regression fitting between transmission rate and inverse film thickness at distinct chill roll temperatures is provided to predict permeation of BOPP produced with over 30 processing scenarios. This shows dominant role of BOPP film thickness and crystallinity in

Oxygen transmission rate. The addition of nucleating agents promotes BOPP crystallinity and barrier performance by 6-8% and up to 8-10% by further thermal annealing for 10 seconds at 20-40 °C below polymer peak melting temperature. Simultaneous addition of nucleating agent and plate-like talc at 4 wt% improves the BOPP Oxygen barrier by 14.5%. Addition of ethylene elastomeric impact modifiers can suppress film crystallinity and barrier properties up to 10% and therefore it is not preferred.

Overall, this research shows that Oxygen permeability of BOPP films can be reduced to 25.9 ± 1.2 (cc.mm/m².day) and Oxygen transmission rate of 319 ± 12 (cc/m².day) by modulating processing parameters and additives used here. The realised Oxygen barrier performance of BOPP is 80% better than commercial coextruded PP-PE films and 80% lower than PET-PE commercial multilayer film used for e.g., fresh salads. It is foreseen that insights and systematic results reported here on sensitivity of BOPP processing scenarios on barrier properties and optimum design window will enhance the technology readiness level (TRL) of monomaterial from two to four. The data can be valuable for both biaxial stretching and blow moulding processes. Further research is needed on compounding different grades of the same base polymer and continuous barrier layer such as EVOH.

Acronyms and abbreviations

BOPP	Biaxially oriented polypropylene film
DfR	Design for recycling
EC	European Commission
ESPR	Ecodesign for Sustainable Products Regulation
MFR	Melt flow rate
OTR	Oxygen transmission rate
PE	Polyethylene polymer
PET	Polyethylene Terephthalate polymer
PP	Polypropylene polymer
PPWR	Packaging and Packaging Waste Regulation
RPM	Revolutions per minute (rpm) rotational speed unit
TRL	Technology readiness level
WP	Work Package

1. Introduction

State-of-the-art flexible plastic food packaging is typically composed of engineered multilayer, multifunctional structures designed to deliver high barrier performance and extend the shelf life of specific food products (Alreshidi et al., 2025). However, the inherent heterogeneity of these multi-material systems presents significant challenges for end-of-life management (Mengozi et al., 2025). Multilayer films are difficult to recycle, as they cannot be readily separated into pure, single-polymer fractions required for efficient polymer-specific sorting and high-quality mechanical recycling. Consequently, such materials are predominantly subjected to downcycling, incineration, or landfilling, leading to diminished material value retention and increased environmental impacts (Anwar et al., 2024).

In response, increasing attention is being directed toward the development of mono-material flexible packaging solutions that maintain the functional performance of conventional multilayer systems while significantly enhancing

recyclability. This transition is strongly driven by evolving regulatory frameworks and circular economy objectives aimed at improving recyclability and increasing material circularity within plastic packaging systems (Stefanini & Vignali, 2026).

The Packaging and Packaging Waste Regulation (PPWR) (Regulation (EU) 2025/40) establishes a harmonised EU framework requiring that all packaging placed on the market be recyclable in an economically viable manner by 2030. This requirement is implemented through a recyclability performance grading system grounded in design-for-recycling criteria, under which packaging is classified into Grade A ($\geq 95\%$ recyclability performance), Grade B ($\geq 80\%$), Grade C ($\geq 70\%$), and a category of technically non-recyclable packaging ($< 70\%$). Within this framework, packaging falling below the 70% recyclability threshold is not permitted on the EU market from 2030 onwards, thereby establishing a minimum recyclability performance requirement across all packaging formats. The regulation is further tightened over time, as Grade C packaging will be phased out from 2038, raising the minimum recyclability compliance level to Grade B ($\geq 80\%$) and accelerating the elimination of low-performing and structurally complex packaging solutions (European Commission, 2025).

Furthermore, Extended Producer Responsibility (EPR) schemes reinforce this transition through eco-modulated fee structures that are directly linked to recyclability performance. Within these schemes, producers are financially incentivised to prioritise packaging designs with higher recyclability grades, such as mono-material structures, as higher-performing packaging is subject to lower fees, while lower-performing packaging incurs higher financial contributions (European Commission, 2025).

Complementing the PPWR, the Ecodesign for Sustainable Products Regulation (ESPR) (Regulation (EU) 2024/1781) establishes upstream product design requirements that integrate circularity, material efficiency, durability, and recyclability across product categories. This regulatory framework promotes simplified material architectures by prioritising design choices that facilitate high-quality recycling, improve sorting efficiency, and enhance secondary raw material recovery. ESPR discourages unnecessary material complexity, including multilayer and multi-material structures that hinder circularity,

thereby reinforcing mono-material design strategies as a preferred approach (European Commission, 2024).

Polypropylene (PP) is a promising material for monomaterial flexible film development due to its high production and collection rates, established recycling and compounding routes, and competitive price compared to e.g., Polyethylene terephthalate (PET). Polypropylene (PP) films can offer low haze and high clarity for aesthetics and excellent water vapour barrier properties (Cigada et al., 2026; Kim et al., 2018). The main bottleneck for monomaterial PP is its poor Oxygen barrier performance which is required for certain food packaging (Kim et al., 2018).

The objective of this work is to increase the technology readiness level (TRL) of monomaterial polypropylene (PP) films from 2 to 4 and to support their further development by systematically identifying key opportunities and limitations. In this context, the study evaluates the influence of film casting and biaxial stretching parameters on the oxygen barrier performance of biaxially oriented polypropylene (BOPP) films, alongside the effect of material modifications, including different PP grades and additives such as talc fillers, nucleating agents, and impact modifiers. By combining controlled processing with material design approaches, the work establishes structure–property relationships that enable the optimisation of monomaterial PP films for high-performance and recyclable food packaging applications, thereby contributing to the transition towards circular plastic packaging systems.

2. Methodology

The overview of research structure is presented in Figure 1 and Figure 2 with two main routes focused on the effect of film processing and different grades of the same base polymer with respect to Oxygen barrier performance.

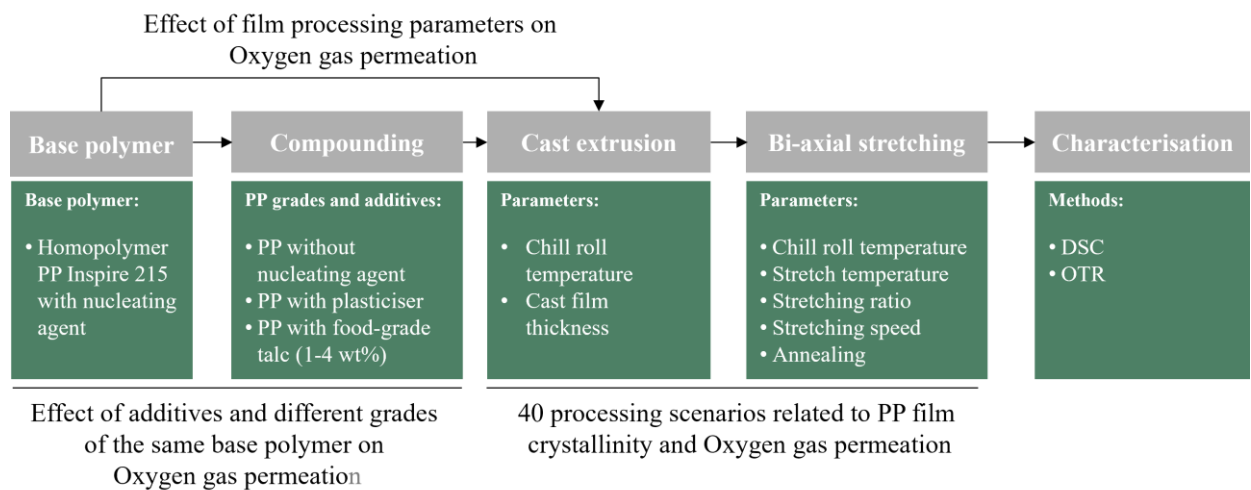


Figure 1. Research structure of report focused on monomaterial film development by tuning processing parameters and additives.

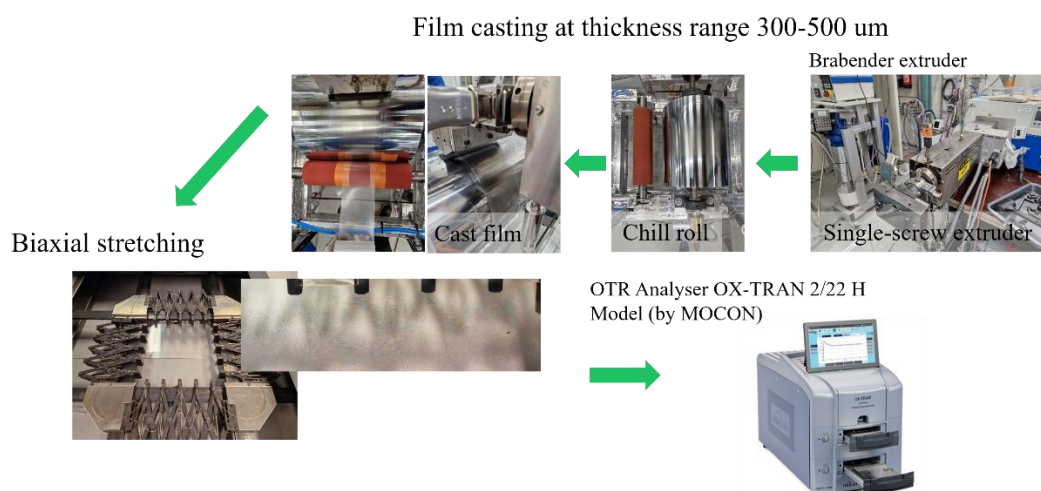


Figure 2. Graphical representation of BOPP development for Oxygen transmission rate testing.

2.1. Materials

The base polymer throughout monomaterial film development was a homopolymer polypropylene (PP) with nucleating agent (PP Inspire 215, Braskem, Germany). The addition of nucleating agent improves the optical clarity of PP. PP Inspire 215 had a Haze of 2.5% (per ASTM D1003) and gloss of 90 (at 45° per ASTM D2457). The clear (i.e., low haze) and high gloss characteristics of this PP grade are suited for food packaging application. The PP Inspire 215 had a melt flow rate (MFR) value of 2.1 (g/10 min) at 230 °C and 2.16 kg conditions reported by the manufacturer per ISO 1133 testing standard. This MFR range makes PP Inspire 215 ideal for sheet extrusion, thermoforming, and injection moulding. The PP Inspire 215 offers an intermediate MFR between film (MFR less than 1 g/10 min) and sheet extrusion grades, therefore, the results can be relevant in both applications.

To investigate effect of nucleating agents on Oxygen permeability of cast and bi-axially oriented PP films, Braskem sourced a research (non-commercial) grade of PP Inspire 215 without any nucleating agent. This grade of material is mentioned as PP without nucleating agent throughout the report.

The contribution of plasticiser on the Oxygen permeability of PP films was studied on plasticised copolymer of PP Inspire 215 base polymer (PP Inspire 137, Braskem, Germany). Based on the manufacturer data sheet, the MFR of PP Inspire 137 is 0.8 (g/10 min) at 230 °C and 2.16 kg conditions, with typical application in blow moulding, film, and profile extrusion. This PP copolymer has haze value of 37% and gloss of 30%, meaning it is not as translucent and clear as the PP homopolymer.

Potential contribution of micro-sized fillers on Oxygen permeability of PP films was investigating by dispersing food contact safe talc. The talc grade was in the form of high aspect ratio lamellar Talc (Stea plus HAR T77, Imerys S. A., Paris, France) compounded with PP Inspire 215 at 1–4 wt% concentration as shown in Figure 3. The PP Talc compounding was realised with a pilot-scale co-rotating twin-screw extruder (ZE25x48D model, Berstorff GmbH, currently operating under KraussMaffei GmbH, Germany) equipped with two separate feeders for

introducing PP pellets and talc fillers. The screw diameter was 25 mm with a process length of 1.2 m. For talc-PP recipe, a 2 kg batch was compounded at a screw speed of 200 (rpm), 0.2 kg/h throughput, and 3 bar pressure. The melt residence time was 18 minutes at 240 °C, while vacuum was applied during the process. The extruder was purged with cleaning material before compounding.

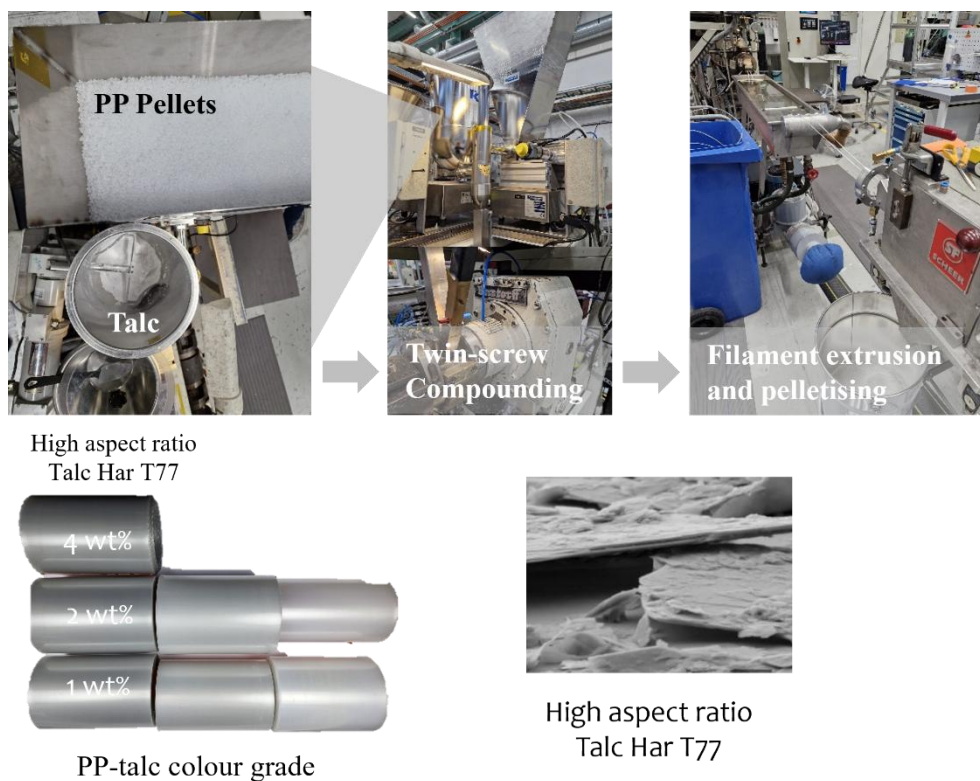


Figure 3. Polypropylene-talc compounding process.

2.2. Cast film extrusion

The cast film extrusion of various PP polymer grades was performed by a single-screw extruder (Plastograph EC, Brabender, 19/25 Z) equipped with a flat die, melt pump, temperature and pressure sensors. The film processing was realised at extrusion rate of 70 (rpm) at 230 °C melt temperature, and throughput of 0.5 meters per minute. The melting temperature of PP 215 homopolymer, used as based polymer this study, was 166 °C, as shown in

Figure 4. The width of flat die was 13 cm, and the width of collected films was 10 cm. The extruded polymer was cast on a chill roll with water circulated control system (Tempro basic C 140, Wittmann). The crystallinity of films was adjusted by varying chill roll temperature from 100 °C to 120 °C. The minimum temperature was selected based on processability (e.g., uniform film formation, and adherence to chill roll). The maximum temperature was selected as close to the 124 °C polymer peak crystallisation temperature as shown in Figure 4, above which films cannot be detached from chill roll. The films were cast at two thickness range of 300 µm and 500 µm by varying the chill roll rotation rate from 8 Hz to 5 Hz, respectively.

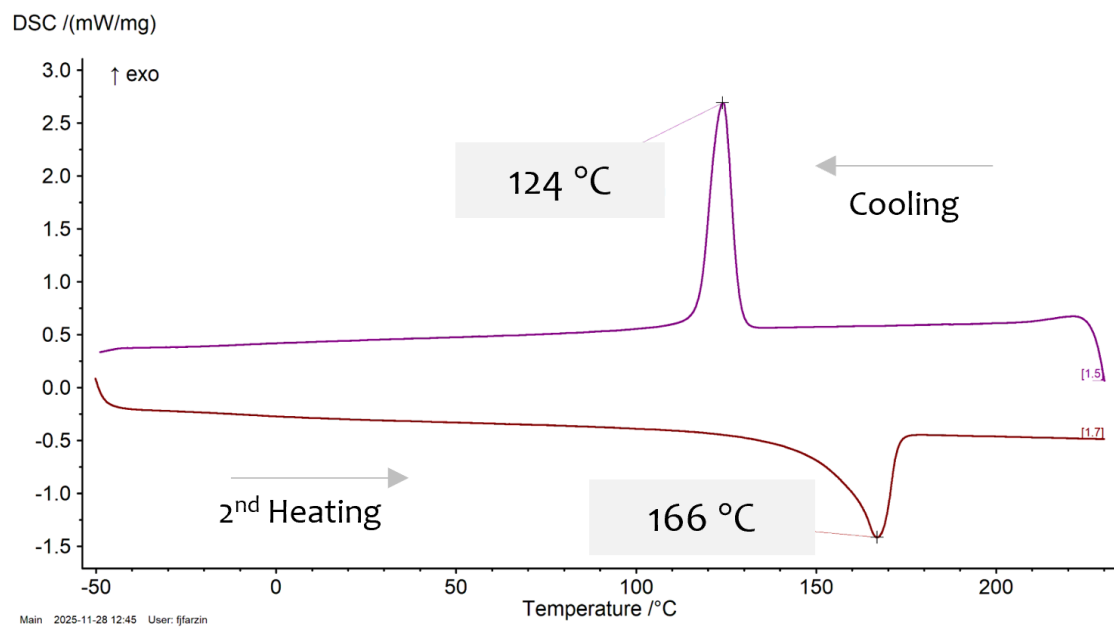


Figure 4. DSC thermogram of PP 215 homopolymer depicting melting and cold crystallisation temperatures.

2.3. Bi-axial film stretching

The PP cast films (300 µm and 500 µm) were biaxially oriented with a simultaneous biaxial stretching device (KARO IV, Brückner Maschinenbau, Siegsdorf, Germany). The stretching was realised at constant displacement rate of 467 (%/second). The objective was to understand contribution of

various stretching parameters on crystallinity and Oxygen Transmission Rate of PP monomaterial. The base polymer for process parameter optimisation was PP homopolymer (PP Inspire 215, Braskem).

2.4. Film crystallinity analysis

The melting and crystallisation behaviour of cast and biaxially oriented PP films was analysed by Differential Scanning Calorimetry (DSC) (Polyma 214, Netzsch, Germany). Tests were performed in Nitrogen atmosphere with a flow rate of 20 mL/min, between -25 °C to 200 °C, at heating rate of 10 °C/min. The method included two dynamic heating cycles and one dynamic cooling cycle, with 5 min isothermal step at the end of each cycle. The polymer melting enthalpy of fusion ΔH_f (J/g) was determined by integration of the first melting peak. The average ΔH_f value from three measurements per series were reported as indication of film crystallinity modulated by various processing conditions and additives. Test samples were punched out of films at 5 mm diameter discs within 5–9 mg weight range.

2.5. Permeability analysis

The barrier properties of monomaterial PP films were analysed in terms of Oxygen Gas Transmission Rate (OTR). The steady-state OTR tests were performed with an ASTM D3985 compliant permeation analyser (OX-TRAN 2/22 H model, MOCON) based on coulometric sensor technology with detection limit of 0.5 to 2000 ($\text{cm}^3 / (\text{m}^2 \cdot \text{day})$). The steady-state Oxygen test gas and carrier Nitrogen gas flow rates were 35 cm^3/min and 80 cm^3/min , respectively. The device was equipped with two independent cells with changeable horizontal test cartridges for measuring parallel samples. Each film sample was prepared in 10 cm × 10 cm (width × length) size and masked to 5.64 cm^2 sample area. The OTR [per $\text{cm}^3 / (\text{m}^2 \cdot \text{day})$] and permeation [per $\text{cm}^3 \cdot \mu\text{m} / (\text{m}^2 \cdot \text{day})$] values were automatically computed by the device computer at 1 atmospheric pressure. The measurements were realised at 23 °C and 50 %RH. Two films were tested per monomaterial series and average values were reported. Test samples were stored in testing environment for one week prior to testing.



3. Results and discussion

The low Oxygen gas barrier properties of polypropylene (PP) is the main bottleneck towards its monomaterial film development. In this context, the effect of all possible processing parameters used for production of cast films and bi-axially oriented films is investigated to understand their contribution to PP barrier properties and recommend an optimum design space. Also, contribution of nucleating agent and talc fillers is investigated.

To provide a perspective on barrier properties of monomaterial BOPP, the corresponding OTR values of state-of-the-art commercial multilayer films are provided in Table 1. The listed multilayer packaging is composed of PET/PE and PP/PE films. In PET/PE films Polyethylene terephthalate (PET) acts as an Oxygen barrier layer and polyethylene (PE) as an adhesive layer and water vapour barrier. In PP/PE films, PP provides rigidity and balanced Oxygen and water vapour barrier properties, while PE is coextruded with a tie layer to PP to facilitate heat sealing for packaging.

3.1. BOPP OTR with respect to film thickness and processing parameters

The Oxygen transmission rate (OTR) of 31 biaxially oriented polypropylene (BOPP) monomaterial films with distinct processing scenarios is summarised in Table 1. The OTR values are listed in an ascending order as function of initial cast film thickness and casting temperature (i.e., chill roll temperature), bi-axial stretching temperature and ratio, annealing, and BOPP film thickness.

Table 1. Summary of Oxygen transmission rate of monomaterial BOPP as a function of processing parameters, listed in a descending OTR order.

Series	Cast film thickness (µm) / Chill roll temperature (°C)	Stretch Temperature (°C) / Stretch ratio	Anneal (°C)	BOPP-Film thickness (µm)	OTR (cc/m ² .day)	Permeation (cc.mm/m ² .day)
OTR	500 µm/120 °C	160/2 x 2	NA	115	319 ± 12	36.0 ± 1.4
43	500 µm/120 °C	165/2 x 2	NA	105	390 ± 15	41.0 ± 1.5
44	500 µm/100 °C	165/2 x 2	NA	116	402 ± 50	46.7 ± 5.2
20	500 µm/100 °C	160/1.5 x 1.5	NA	242	412 ± 10	99.7 ± 0.4
42	500 µm/120 °C	165/3 x 3	NA	51	565 ± 32	28.8 ± 1.6
21	500 µm/100 °C	160/2 x 2	NA	118	579 ± 74	68.3 ± 8.7
40	500 µm/120 °C	160/3 x 3	NA	48	587 ± 74	28.2 ± 3.6
27	300 µm/120 °C	165/2 x 2	NA	53	614 ± 49	32.5 ± 2.6
45	500 µm/100 °C	165/3 x 3	NA	42	758 ± 55	31.8 ± 2.3
46	300 µm/100 °C	160/2.5 x 2.5	NA	38	764 ± 11	29.1 ± 0.4
22	500 µm/100 °C	160/2.5 x 2.5	NA	75	978 ± 147	73.5 ± 11.1
52-8	500 µm/120 °C	150/3.5 x 3.5	NA	35	1068 ± 25	37.4 ± 0.8
26	300 µm/120 °C	160/2 x 2	NA	53	1109 ± 10	58.7 ± 0.6
52-7	500 µm/120 °C	155/3.5 x 3.5	NA	28	1189 ± 95	38.6 ± 1.2
48	300 µm/120 °C	165/2.5 x 2.5	NA	30	1193 ± 200	35.8 ± 9.3
62-2	500 µm/120 °C	160/3.5 x 3.5	130 C, 10s	28	1204 ± 13	33.7 ± 0.4
52-5	500 µm/120 °C	160/3.5 x 3.5	130 C, 10s	31	1234 ± 22	38.1 ± 4.4
47	300 µm/100 °C	165/2.5 x 2.5	NA	29	1249 ± 340	36.2 ± 10.1
23	500 µm/100 °C	160/3 x 3	NA	49	1251 ± 62	61.3 ± 3.1
52-1	500 µm/120 °C	160/3.5 x 3.5	NA	29	1256 ± 2	36.8 ± 1.3
62-1	500 µm/120 °C	160/3.5 x 3.5	NA	28	1281 ± 13	35.9 ± 0.4
52-4	500 µm/120 °C	160/3.5 x 3.5	120 C, 10s	28	1281 ± 29	36.5 ± 0.1
52-6	500 µm/120 °C	160/3.5 x 3.5	140 C, 10s	28	1296 ± 1	36.3 ± 0.1
57-2	500 µm/120 °C	160/3.5 x 3.5	130 C, 10s	28	1367 ± 37	38.3 ± 1.1
52-3	500 µm/120 °C	160/3.5 x 3.5s	NA	28	1368 ± 81	37.5 ± 0.7
57-1	500 µm/120 °C	160/3.5 x 3.5	NA	28	1386 ± 75	39.4 ± 0.8
25	300 µm/120 °C	160/2.5 x 2.5	NA	47	1423 ± 200	66.8 ± 11.4
41	300 µm/120 °C	165/3 x 3	NA	20	1443 ± 148	28.9 ± 2.9
37	300 µm/120 °C	160/3 x 3	NA	27	1476 ± 150	39.8 ± 4.1
52-2	500 µm/120 °C	160/4 x 4	NA	23	1651 ± 60	37.9 ± 1.4
24	500 µm/100 °C	160/3.5 x 3.5	NA	32	2083 ± 40	66.7 ± 0.4
Commercial PET-PE (Meat-Chicken)				60	3 ± 0.1	0.2 ± 0.05
Commercial PET-PE (Salad-Chicken)				52	79 ± 1	4.1 ± 0.1
Commercial PP-PE (LPEEL)				50	2975 ± 73	148.7 ± 3.7
All series are based on PP homopolymer with nucleating agent. Series with blue shade do not include nucleating agent, and series in grey include nucleating agent and 4 wt% talc.						

The sensitivity of BOPP OTR on processing parameters calculated by Pearson linear correlation coefficient is summarised in Table 2. The Pearson correlation is linear and interconnection between parameters is not considered. Based on the results, the Oxygen barrier properties of BOPP films are strongly sensitive to the film thick value (i.e. and stretch ratio as it is directly related to thickness), followed by chill roll cast temperature, initial cast film thickness, and stretch temperature.

Table 2. Sensitivity of BOPP Oxygen transmission rate (OTR) to film processing parameters per Pearson linear correlation calculation.

Parameters	Correlation	Sensitivity
Stretch ratio	+0.75	Strong
Stretch temperature	-0.15	Weak
BOPP film thickness	-0.70	Strong
Cast film thickness	-0.25	weak
Chill roll cast temperature	+0.30	Moderate

The negative Pearson correlation sign in Table 2, for instance in case of film thickness means increasing that processing parameter will reduce the OTR resulting in better Oxygen barrier performance. The OTR of BOPP films presented in Table 1, is plotted in Figure 5 with respect to their thickness. The overall trend shows that OTR is inversely proportional to film thickness governed by Fick's first law of diffusion under steady-state conditions.

The best barrier performance (i.e., lowest OTR) for BOPP in Figure 5 is in the range of 300–400 (cc/m².day) corresponding to cast film thickness of 500 µm, chill roll temperature of 100–120 °C, stretch temperature of 160–165 °C, and low stretch ratio of 1.5 x 1.5 and 2 x 2. The average thickness values of BOPP films processed with those parameters are in the range of 100–115 µm.

Based on the results the best compromise between film thickness and good barrier properties (i.e., low OTR) is in the range of 50 µm with OTR of 565 ± 32 (cc/m².day), for instance film series 42 in Table 1, produced with 3 x 3 stretch ratio and stretch temperature of 165 °C. At similar film thickness of 50 µm, OTR of BOPP (3 x 3, 165 °C) 565 ± 32 (cc/m².day) is 80% lower than commercial coextruded PP–PE LPEEL film. At similar thickness range, OTR of BOPP (3 x 3, 165

°C) 565 ± 32 (cc/m².day) is 80% higher compared to OTR of state-of-the-art multilayer PET/PE 79 ± 1 (cc/m².day). According to the data in Figure 5, the reduction of OTR as a function of film thickness slows in the range of 100–115 µm above which there is no meaningful improvement in barrier properties.

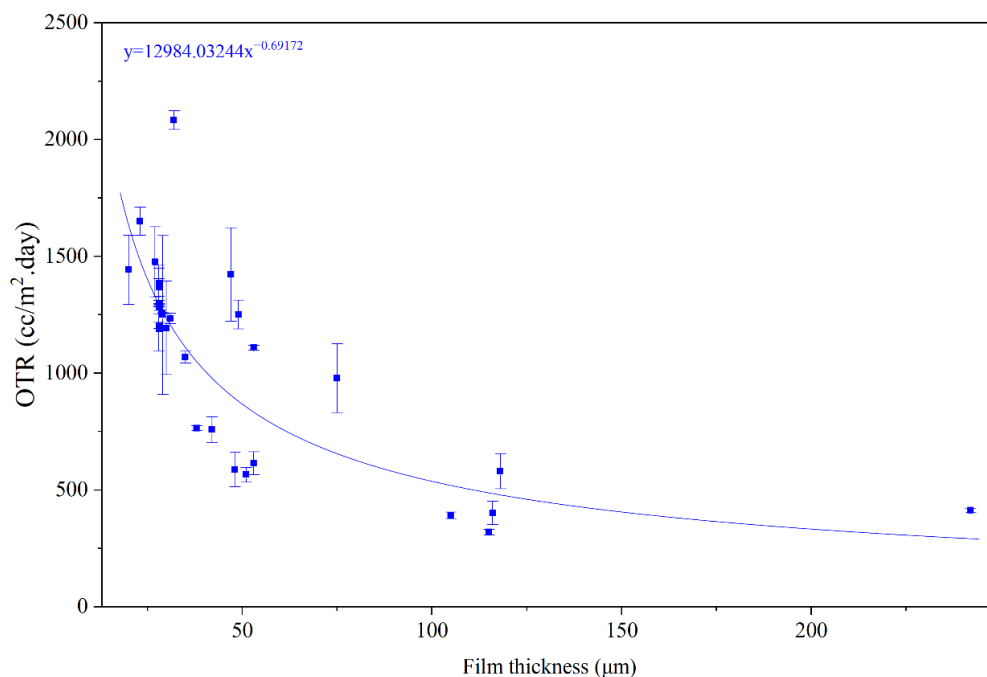


Figure 5. Oxygen transmission rate as a function of BOPP film thickness.

3.2. BOPP Oxygen permeation adjusted by processing parameters

The Oxygen barrier performance of BOPP films presented in Figure 5, largely depend on film thickness but also other processing parameters which control film thickness and film crystallinity. To separate contribution of processing parameters and their impact on barrier properties, it is necessary to focus on Oxygen permeation as an intrinsic property. Permeation is film OTR multiplied by its thickness. The average Oxygen permeation of BOPP films is listed in Table 3 in descending order as a function of 31 distinct processing scenarios.

Table 3. Oxygen permeability of BOPP as a function of processing parameters, listed in a descending Oxygen permeation order.

Series	Cast film thickness (µm) / Chill roll temperature (°C)	Stretch temperature (°C) / Stretch ratio	Anneal (°C)	Enthalpy of fusion (J/g)	BOPP-Film thickness (µm)	Permeation (cc.mm/m ² .day)
51	500 µm/120 °C	165/3 x 3	NA	116	30	25.9 ± 1.2
40	500 µm/120 °C	160/3 x 3	NA	115	48	28.2 ± 3.6
42	500 µm/120 °C	165/3 x 3	NA	114	51	28.8 ± 1.6
41	300 µm/120 °C	165/3 x 3	NA	114	20	28.9 ± 2.9
46	300 µm/100 °C	160/2.5 x 2.5	NA	113	38	29.1 ± 0.4
45	500 µm/100 °C	165/3 x 3	NA	115	42	31.8 ± 2.3
27	300 µm/120 °C	165/2 x 2	NA	113	53	32.5 ± 2.6
62-2	500 µm/120 °C	160/3.5 x 3.5	130 C, 10s	105	28	33.7 ± 0.4
48	300 µm/120 °C	165/2.5 x 2.5	NA	114	30	35.8 ± 9.3
62-1	500 µm/120 °C	160/3.5 x 3.5	NA	104	28	35.9 ± 0.4
39	500 µm/120 °C	160/2 x 2	NA	113	115	36.0 ± 1.4
47	300 µm/100 °C	165/2.5 x 2.5	NA	113	29	36.2 ± 10.1
52-6	500 µm/120 °C	160/3.5 x 3.5	130 C, 10s	111	28	36.3 ± 0.1
52-4	500 µm/120 °C	160/3.5 x 3.5	120 C, 10s	113	28	36.5 ± 0.1
52-1	500 µm/120 °C	160/3.5 x 3.5	NA	113	29	36.8 ± 1.3
52-8	500 µm/120 °C	150/3.5 x 3.5	NA	101	35	37.4 ± 0.8
52-3	500 µm/120 °C	160/3.5 x 3.5s	NA	110	28	37.5 ± 0.7
52-2	500 µm/120 °C	160/4 x 4	NA	110	23	37.9 ± 1.4
52-5	500 µm/120 °C	160/3.5 x 3.5	110 C, 10s	107	31	38.1 ± 4.4
57-2	500 µm/120 °C	160/3.5 x 3.5	130 C, 10s	108	28	38.3 ± 1.1
52-7	500 µm/120 °C	155/3.5 x 3.5	NA	100	28	38.6 ± 1.2
57-1	500 µm/120 °C	160/3.5 x 3.5	NA	102	28	39.4 ± 0.8
37	300 µm/120 °C	160/3 x 3	NA	100	27	39.8 ± 4.1
43	500 µm/120 °C	165/2 x 2	NA	115	105	41.0 ± 1.5
44	500 µm/100 °C	165/2 x 2	NA	115	116	46.7 ± 5.2
26	300 µm/120 °C	160/2 x 2	NA	115	53	58.7 ± 0.6
23	500 µm/100 °C	160/3 x 3	NA	113	49	61.3 ± 3.1
24	500 µm/100 °C	160/3.5 x 3.5	NA	111	32	66.7 ± 0.4
25	300 µm/120 °C	160/2.5 x 2.5	NA	114	47	66.8 ± 11.4
21	500 µm/100 °C	160/2 x 2	NA	113	118	68.3 ± 8.7
22	500 µm/100 °C	160/2.5 x 2.5	NA	107	75	73.5 ± 11.1
20	500 µm/100 °C	160/1.5 x 1.5	NA	110	242	99.7 ± 0.4
Commercial PET-PE (Meat-Chicken)					60	0.2 ± 0.05
Commercial PET-PE (Salad-Chicken)					52	4.1 ± 0.1
Commercial PP-PE (LPEEL)					50	148.7 ± 3.7

The sensitivity of BOPP permeation values reported in Table 3 to each processing parameter is calculated by Pearson linear correlation coefficient and listed in Table 4. Based on permeation results, the most critical processing

parameters for modulating Oxygen barrier performance of BOPP are chill roll cast temperature, and stretch ratio, followed by less sensitive parameters stretch temperature and cast film thickness. All these parameters promote film crystallinity as reflected in enthalpy of fusion values in Table 3. Polymer crystalline fraction acts as Oxygen impermeable phase.

Table 4. Sensitivity of BOPP intrinsic permeation to film processing parameters per Pearson linear correlation calculation.

Parameters	Correlation	Sensitivity
Chill roll cast temperature	-0.65	Strong
Stretch ratio	-0.55	Strong
Stretch temperature	-0.25	Weak
Cast film thickness	0.15	Weak

By realising the importance of chill roll temperature on BOPP film crystallinity and permeation based on sensitivity analysis in Table 4, it is beneficial to analyse permeation variance at different chill roll temperatures for its statistical significance and predictive regression models.

The OTR of BOPP films processed at different stretch temperatures and ratios from Table 3 is plotted as a function of inverse film thickness in Figure 6. The data fitting slope of OTR and inverse thickness represents film permeation based on diffusion theory. Separate linear regression models are fitted to BOPP films processed at low and high chill roll temperatures to signify its contribution on Oxygen permeation. The analysis shows that by increasing chill roll temperatures from 100 °C to 120 °C the BOPP permeation decrease by 40% (from 60.841 to 35.072 cc.mm/m².day), meaning better barrier performance. The linear regression provides a predictive permeation model for BOPP films processed at different chill roll temperatures.

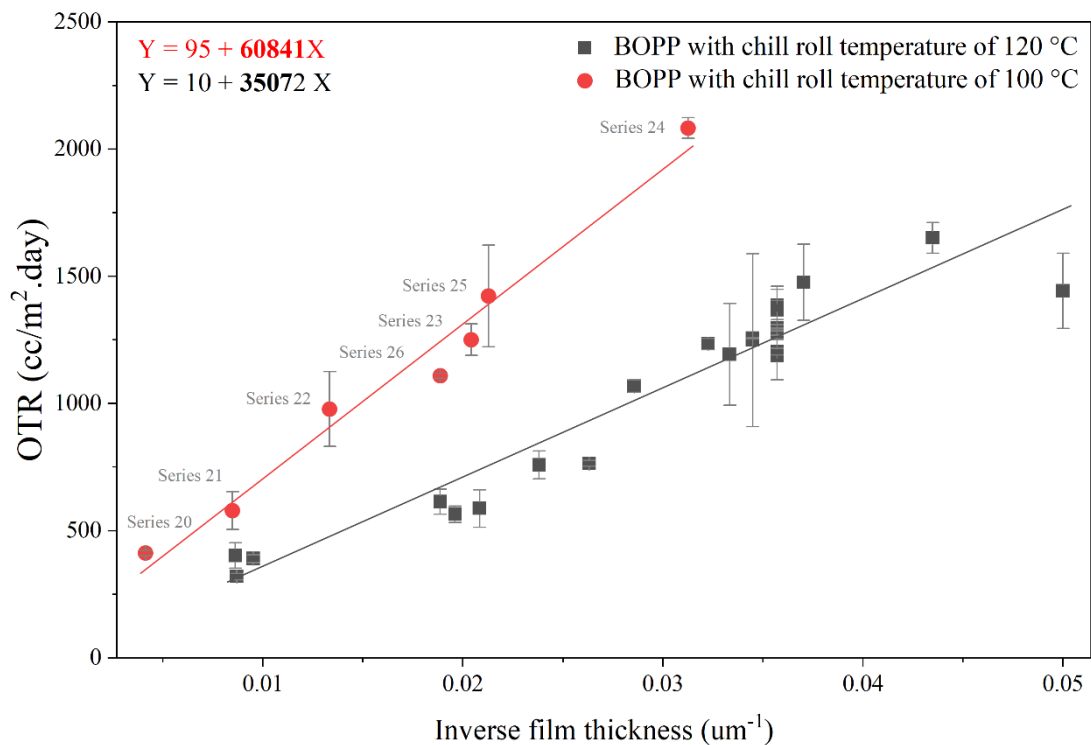


Figure 6. BOPP OTR - inverse film thickness plot with their linear fit representing Oxygen permeation.

The Oxygen permeation of PP films processed at low and high chill roll temperatures is presented in Figure 7 as a function of film thickness and stretch ratio. The analysis is based on full factorial design demonstrating interaction between processing parameters. The Oxygen permeation of cast PP film (homopolymer with nucleating agent) is equally sensitive to film thickness and chill roll temperature factors at both high and low levels. For instance, Oxygen permeation of a 300 µm thick PP casted at 100 °C (151 cc.mm/m².day) reduces by 53% and 55% with either maximising its thickness or cast temperature.

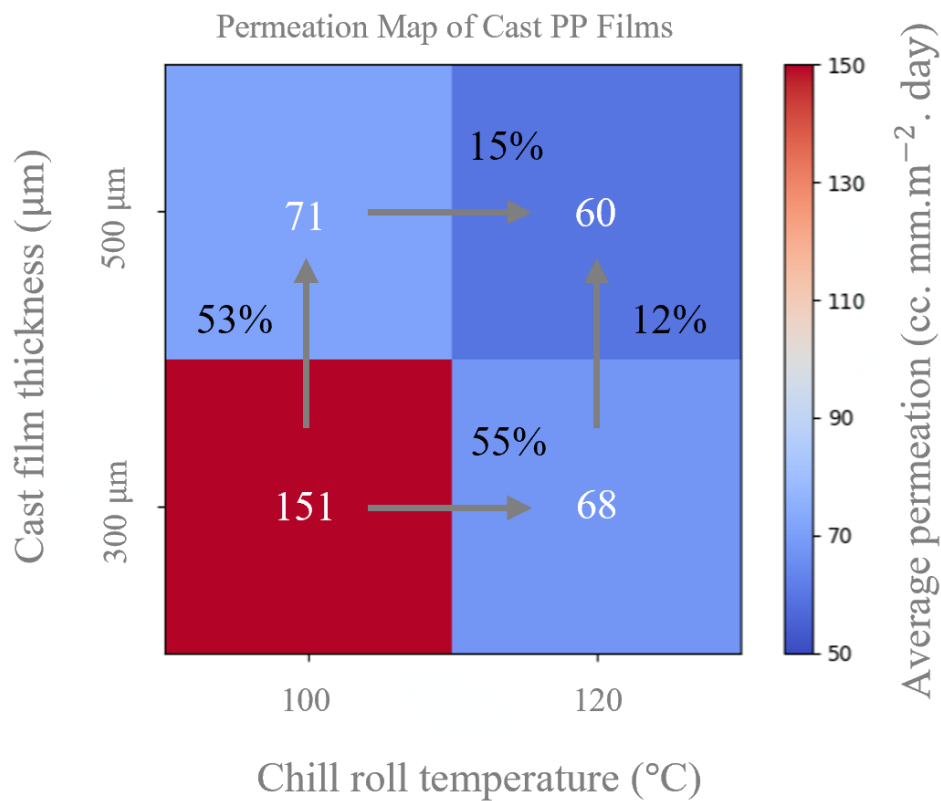


Figure 7. Sensitivity of cast film permeation to chill roll temperature.

The contribution of chill roll temperature to better barrier performance in Figure 7 is based on higher film crystallinity promoted by temperature increase. At either low or high film thickness range, increasing chill roll temperature from 100 °C to 120 °C (near peak crystallisation temperature) enhances film crystallinity by 8-9% reflected in fusion enthalpy values in Table 5. However, at similar chill roll temperature of 100 °C or 120 °C, enhancing film thickness from 300 μm to 500 μm has a minor impact on enthalpy of fusion reflective of film crystallinity. Results suggest that contribution of cast film thickness to Oxygen permeation is mainly due to large diffusion path as physical barrier rather than enhanced crystallinity.

Table 5. Oxygen permeation and fusion enthalpy of cast PP film in response to two level full factorial design with film thickness and chill roll temperature as processing parameters (i.e., factors).

Film thickness (μm)	Chill roll temperature ($^{\circ}\text{C}$)	Enthalpy of fusion (J/g)	Permeation ($\text{cc.mm/m}^2.\text{day}$)
500	120	112	59.53 ± 0.01
500	100	102	71.20 ± 0.01
300	120	109	67.68 ± 0.01
300	100	101	150.89 ± 0.01

The BOPP Oxygen permeation is presented by two level full factorial design as a function of stretch ratio and chill roll temperature in Figure 8. A blank permeation value (e.g., in case of 2 x 2, 100 $^{\circ}\text{C}$, 300 μm) means that Oxygen transmission was so high that stabilisation did not realise and data was not capture, so OTR was larger than detection limit. According to the results, increasing chill roll temperature of cast films from 100 $^{\circ}\text{C}$ to 120 $^{\circ}\text{C}$ reduce the average Oxygen permeation of BOPP films by 47% for 2 x 2 and 54% for 3 x 3 stretch ratio. Contribution of stretching ratio on BOPP film permeation is interconnected to chill roll temperature (i.e., crystallinity of film before biaxial stretching). At 100 $^{\circ}\text{C}$ chill roll temperature for 500 μm thick films, 3 x 3 stretching has 10% lower permeation compared to 2 x 2 case, while the difference is 22% for films casted near peak crystallisation temperature of 120 $^{\circ}\text{C}$. Interestingly, the results show the significant importance of starting cast film crystallinity and processing history on permeation and barrier performance of biaxially oriented monomaterial films.

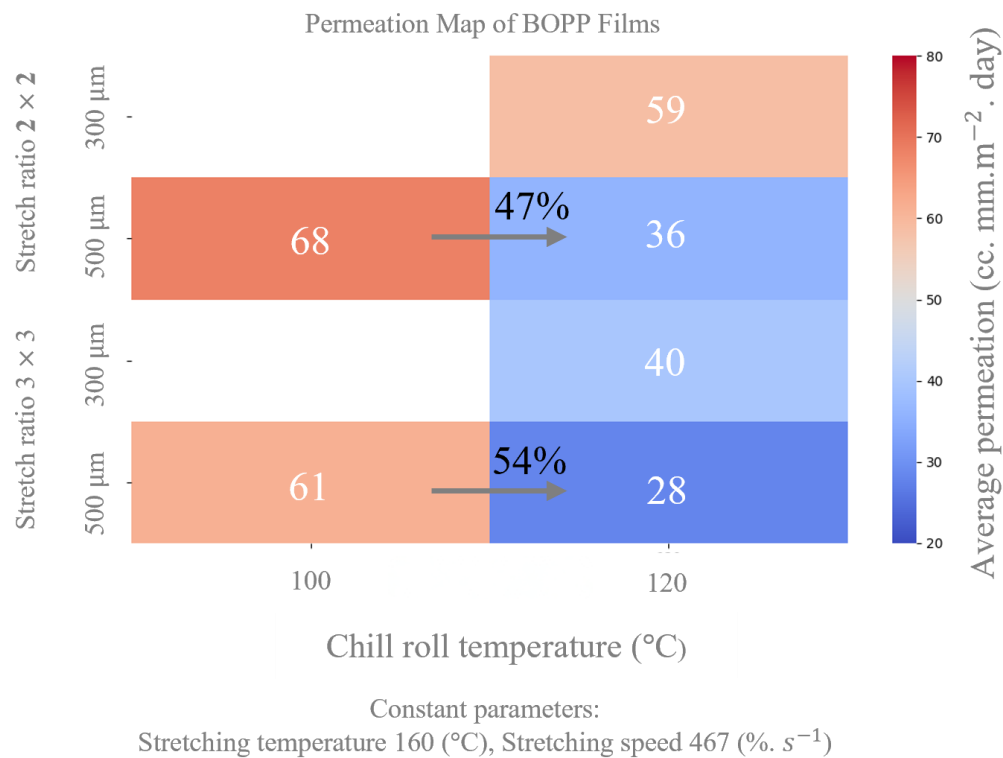


Figure 8. Permeation of BOPP films as a function of chill roll temperature and stretch ratio.

3.3. Effect of additives and annealing

The Oxygen permeation analysis of BOPP with respect to processing parameters indicated high sensitivity to film crystallinity controlled by its thermal history (i.e., chill roll temperature) and stretching ratio with complimentary contribution of stretching temperature. Here, we investigate effect of nucleating agent, plate-like talc additives, elastomeric impact modifiers, and thermal annealing on film crystallinity and barrier properties.

The Oxygen permeability and crystallinity of cast homopolymer PP films with and without elastomeric impact modifier is presented in Table 6. The cold crystallisation peak of impact modified PP is at 116 °C compared PP with crystallisation peak at 124 °C. Therefore, the chill roll temperature which has the highest contribution to Oxygen barrier properties can be set to maximum

temperature of 110 °C for plasticised PP compared to 120 °C for the reference PP due to processing limitations. As a result, the ultimate Oxygen barrier performance of impact modified PP at 110 °C is 10% lower than the reference PP casted at 120 °C. The inferior barrier performance of impact modified PP can be justified by its lower degree of crystallinity compared to the reference grade reflected in enthalpy of fusion values. At lower casting temperatures of 90–100 °C the impact modified PP present better barriers performance compared to reference PP. The difference can be because at similar chill roll temperature (e.g., 100 °C or 110 °C) the impact modified PP is always casted closer to its peak crystallisation of 116 °C compared to reference PP. This might ensure better molecular packaging and crystallinity for impact modified PP at lower cast temperatures. A further investigation would be needed to understand contribution of impact modifiers in BOPP films.

Table 6. Effect of elastomeric impact modifier on Oxygen permeability of cast film PP with 300 µm thickness.

Type of additive and focus	Chill roll temperature (°C)	Cold crystallisation peak (°C)	Enthalpy of fusion (J/g)	Permeation (cc.mm/m ² .day)
PP homopolymer with impact modifier	90	116	81	82.1
	100		83	79.9
	110		84	75.7
PP homopolymer with nucleating agent	100	124	103	151.1
	110		107	137.6
	120		110	67.8

The Oxygen permeability of BOPP films with nucleating agent and talc additives coupled with thermal annealing is provided in Table 7. All BOPPs in Table 8 have film thickness of 28 µm, produced at biaxial simultaneous stretching ratio of 3.5 x 3.5 at 160 °C. Based on the results, introducing nucleating agent to homopolymer BOPP enhance its crystallinity (i.e., enthalpy

of fusion) and Oxygen barrier properties by 9.7% and 6.6% respectively. The contribution of nucleating agent is further enhanced by thermal annealing of BOPP films at 110–130 °C range for 10 seconds. Annealing at 130 °C enhances the barrier performance of BOPP with nucleating agent up to 7.8% compared to 2.8% for BOPP without nucleating agent. The simultaneously stretched BOPP film has slightly higher crystallinity compared to Step* BOPP produced by step-by-step stretching while they have comparable Oxygen permeability (36.8 ± 1.3 for simultaneous stretching and 37.5 ± 0.7 for step-by-step stretching). Simultaneous addition of nucleating agent and 4 wt% plate-like talc improves the BOPP barrier performance by 8.8% (net talc contribution 2.4%). Further annealing of BOPP with nucleating agent and 4wt% talc increases the Oxygen barrier performance by maximum rate of 14.5% (net talc contribution 8.4%). This can be due to the fact that talc creates new crystal nucleation sites for enhanced crystallinity and tortuous Oxygen diffusion path. Further research with X-ray diffraction is needed in future to fully understand contribution of fillers and processing parameters in film crystallinity.

Overall, the results provide insights on contribution of nucleating agents up to 6–8% in Oxygen barrier performance of monomaterial BOPP which can be further increase to 8–10% by further thermal annealing. Further research on higher concentration of talc or different additives can be beneficial. However, it should be noted that addition of fillers at higher loading than 4 wt% or annealing can adjust the haze and transparency of flexible packaging when desired.

Table 7. Effect of nucleating agent, plate-like talc additives, and annealing on Oxygen permeation of BOPP films.

Type of additive and focus	Anneal (°C)	Enthalpy of fusion (J/g)	Permeation (cc.mm/m ² .day)	Improvement
BOPP no nucleating agent	NA	102	39.4 ± 0.8	Reference
	130	108	38.3 ± 1.1	2.8%
BOPP with nucleating agent	NA	113	36.8 ± 1.3	6.6%
	110	107	38.1 ± 4.4	3.3%
	120	113	36.5 ± 0.1	7.4%
	130	111	36.3 ± 0.1	7.8%
Step* BOPP with nucleating agent	NA	110	37.5 ± 0.7	4.8%
BOPP with 4 wt% and nucleating agent	NA	104	35.9 ± 0.4	8.8%
	130	105	33.7 ± 0.4	14.5%

Constant processing parameters for BOPP production:

All BOPPs has thickness of 28 µm, stretching temperature of 160 °C, and simultaneous stretching ratio of 3.5 x 3.5. The Step* BOPP is produced via step-by-step stretching.

4. Conclusions and recommendations

This deliverable demonstrates an engineering pathway to design monomaterial food packaging at TRL 2-4 by modulating film thickness, processing parameters, and additives which do not impede recycling. It is driven by the Packaging and Packaging Waste Regulation (PPWR) to ban non-recyclable multilayer packaging from EU market by 2030. The research objective is to improve low Oxygen barrier properties of monomaterial polypropylene (PP) films for shelf-life protection. Focus is to systematically investigate distinct effect of cast film and biaxial stretching parameters and their synergetic contribution on barrier properties based on full factorial design and Pearson correlation coefficient for sensitivity analysis. Analysis covers all processing scenarios concerning biaxially oriented polypropylene (BOPP). The key insights and findings are following:

- The BOPP Oxygen barrier properties are extremely sensitive to film thickness by 70% compared to 30% for all other parameters. The best compromise between film thickness and Oxygen transmission rate (OTR) is realised in thickness range of 40-50 μm with 500-600 OTR ($\text{cc}/\text{m}^2\cdot\text{day}$). The minimum OTR of 300-400 ($\text{cc}/\text{m}^2\cdot\text{day}$) is realised at thickness range of 100-120 μm identified as a physical limit beyond which no improvement in barrier properties is achieved.
- The best Oxygen barrier performance of monomaterial BOPP per process optimisation is 80% higher than commercial PE-PE and 80% inferior to state-of-the-art PE-PET at 50 μm film thickness. For OTR in same level as PE-PET our results provide valuable insights on process optimisation, but further research on compounding and additives is needed at higher TRL.
- The Oxygen permeation analysis of BOPP processed with over 30 distinct scenarios shows highest sensitivity to cast film crystallinity before biaxial stretching. The cast film crystallinity dominantly depends on chill roll (i.e., casting) temperature. Stretching ratio and temperature have second and third level of importance for BOPP permeation.

- The full factorial parametric analysis highlights synergetic effect of stretching ratio and film thermal history. The contribution of chill roll temperature is more pronounced at higher stretch ratios.
- Film crystallinity has minor sensitivity to thickness compared to casting and stretching temperatures.
- Based on the results, a linear regression model of OTR and inverse film thickness grouped at distinct chill roll temperatures can predict Oxygen permeation of BOPPs processed with various stretching scenarios. This highlights dominant contribution of film thickness and chill roll temperature on barrier properties. Overview of parametric sensitivities is presented in Figure 9.
- Nucleating agents improve BOPP crystallinity and Oxygen barrier properties up to 6-8% and up to 8-10% by further thermal annealing for 10 seconds at 20-40 °C below polymer peak melting temperature. Addition of plate-like talc at 4 wt% provides additional crystal nucleating agents and enhance the BOPP barrier performance by 14.5%.
- To maximise BOPP Oxygen barrier properties, it is recommended to cast film near peak crystallisation temperature, ensure film thickness in the range of 40-100 µm. To realise this stretching ratio shall be within 3-4 dependant of cast film thickness. Stretching temperature can be at peak melting temperature and further annealed at 20 °C below stretching temperature for less than a minute (e.g., 10 seconds for industry relevance).

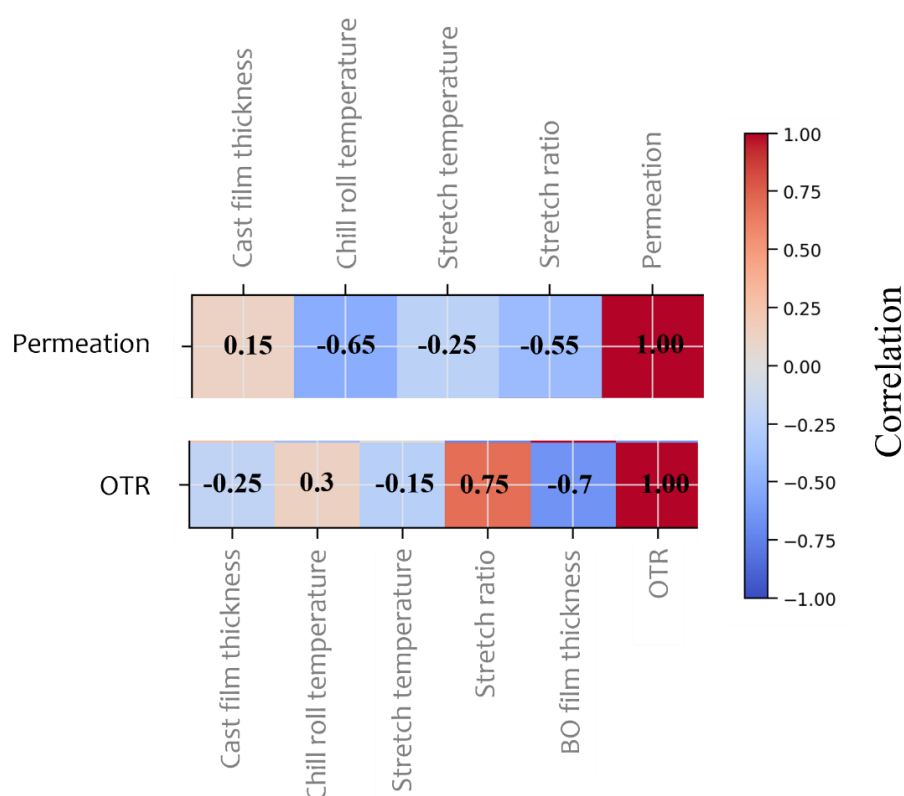


Figure 9. Sensitivity of BOPP barrier properties to processing parameters.

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RecyClass assesses the recyclability of a plastic package providing a ranking from A to F. RecyClass also provides specific indications and recommendations on how to improve packaging design to fit current recycling technologies. More information at <https://recyclclass.eu/>

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