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A Study Into the Hot Tack and Cooled Seal Performance of Emerging Coated Papers for Primary Flexible Food Packaging

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Keywords: coated paper | flexible food packaging | form-fill-sealing | hot tack | seal strength

ABSTRACT

In response to the need to employ recyclable materials for food packaging, the resurgence of paper as a primary flexible packaging material is driven by consumer trust in paper and its renewable wood fibre composition, strengthened by a well-established recycling infrastructure. A diverse range of coated papers and coatings has become accessible in the market, specifically tailored for applications in horizontal and vertical form-fill sealing. Within the framework of the CORNET-TETRA project HBC.2021.0288 REPAC², a careful selection of 16 food-grade coated papers and/or coatings, slated for introduction to the market either presently or within the next 2 years, has been undertaken. This study focused on evaluating the processing window for superior hot tack and cooled seal performance of coated papers, in relation with the composition of paper coatings as identified using Fourier transform infrared (FTIR) measurements, and their thermal softening or melting characteristics as identified through differential scanning calorimetry (DSC). Thermal behaviour in DSC thermograms is used to categorize the commercial coated papers into distinct classes. Class I papers, mainly with acrylic-based coatings, exhibit high hot tack strengths (0.2–0.7 N/mm) and a glass transition temperature (T_g) close to the seal initiation temperature but do not considerably gain strength with further cooling. Class II papers are thermally inert in DSC with minimal thermoplastics, leading to weak seals. In contrast, classes III and IV, including wax-based and polyolefin-based or polyvinylalcohol-based (PVOH) coatings, respectively, show low initial hot tack strengths that considerably increase upon adequate cooling. Particularly, the Class IV papers with polyolefin-based coatings have well-performing seals. Despite having relatively low hot tack strengths after cool times of 0.1 s, below 0.3 N/mm, high strengths can be obtained after adequate cooling with outliers reaching 0.74 and 1.14 N/mm. Additionally, the influences of seal parameters on seal performance were evaluated. The study reveals that cool time, seal pressure and, to a lesser extent, seal time significantly impact hot tack strength, consistent with prior research. The critical role of jaw temperature in heat conductive sealing is affirmed, as it dictates the efficacy of other seal parameters. Seal initiation occurs at 75°C for four papers, and nine others necessitate temperatures equal to or exceeding 100°C, with one paper only displaying seal initiation at 195°C. This variation highlights the requisite for tailored temperature windows for effective sealing of these papers. As such, deeper insights into the intricate interplay between coating composition, thermal properties and seal performance are obtained in order to support advances in sustainable packaging technology.

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1 | Introduction

In the field of primary flexible food packaging, paper is experiencing a resurgence as material, largely due to its long-standing record of effective recycling. Paper is extensively recycled, constituting half of the global materials collected for recycling in 2021 [1]. Additionally, the renewable nature of paper fibres and the perception of paper as a sustainable packaging material by consumers further enhance its appeal as a viable option in this sector. To incorporate paper into form-fill-seal lines currently used for producing plastic bags, it must be made heat-sealable. Because paper is not inherently heat-sealable, a thermoplastic layer is added [2]. However, the resulting coated or laminated papers face several limitations. The first is the permissible thickness of the plastic layer, which varies across countries, for recycling in standard mills. A recent harmonized European protocol from 4evergreen states that packaging papers are negatively scored and, therefore, not considered recyclable in a standard mill if more than 20% of rejects are found after repulping [3]. Given the narrow thickness range of 40–100 μm for papers used in flexible applications, the maximum allowable thicknesses will be considerably under 20 μm . Furthermore, sealing is not the only requirement for this type of packaging; ensuring sufficient gas and moisture barriers, often achieved with plastic or metal layers, is also critical. A second limitation is paper's low thermal conductivity compared to plastic [4]. A third limitation is paper's low dead fold, leading to higher spring-back force when seal jaws are opened. This combination of limitations can adversely affect seal integrity in form-fill-sealing. Sufficient flow of thermoplastic material is needed to fill or caulk voids, in particular at the intersection of vertical and cross-seals [5]. The high spring-back force may compromise the seal during the opening of the seal jaws, whereas suboptimal thermal conductivity can inhibit efficient cooling, increasing the risk of leaks in sealed bags. To evaluate seal performance, hot tack tests are instrumental in measuring the strength immediately after the hot seal jaws are opened, allowing for only minimal cooling. This provides critical insights into the effectiveness of the seal under these challenging conditions. Additionally, achieving high seal strength post-cooling is crucial for subsequent processes after packaging, such as storage, transportation and handling in stores, to ensure leak-tight delivery to the end consumer. In the CORNET project 'REPAC2', 16 materials considered for the European market within 2 years were selected [6]. The seal strength of coated papers was determined after very brief and long cool times. These data are crucial to assess if these materials can be considered in form-fill-seal lines, currently used for full plastic bags. The objective of this study is to analyse the seal performance of commercial coated papers by correlating seal outcomes with the coating composition and thermal properties and by evaluating the influences of various seal parameters, including jaw temperature, seal time, seal pressure and cool time.

2 | Materials and Methods

2.1 | Materials

In this study, 16 food-grade heat-sealable materials selected for their relevance in the European market are analysed. These materials vary in terms of barrier properties, production processes

and polymer origins in the coatings. Due to the confidentiality inherent in commercial research, the composition details of the papers and coatings were largely undisclosed. To uphold confidentiality in projects involving commercial materials, reliance was exclusively placed on descriptions provided by the companies, complemented by validation through attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR) against a comprehensive library, ensuring accuracy and reliability of the descriptions. Among the coated papers, 12 feature dispersion coatings, two are coated by extrusion, and two possess a wax coating. Although 14 of these materials are existing commercial coated papers, the papers with codes II.a and IV.a (as indicated in Table 1) were specially coated in a laboratory setting. This selection represents the diversity of packaging materials and serves as a basis for analysing seal performance.

All papers were stored for a minimum of 48 h in a standard atmosphere, as described in ISO 187 [7], at $23^{\circ}\text{C} \pm 1^{\circ}\text{C}$ and $50\% \pm 2\%$ relative humidity, with subsequent methods conducted in this atmosphere.

2.2 | Methods

2.2.1 | Coating

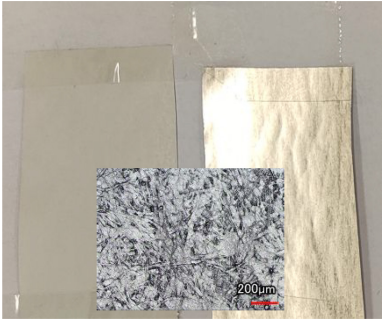
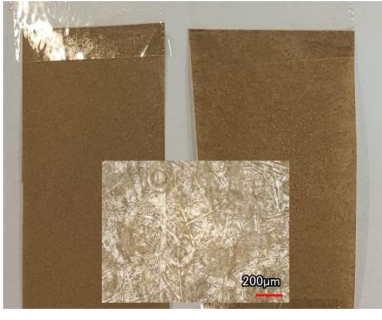
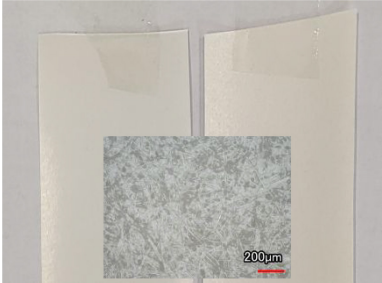
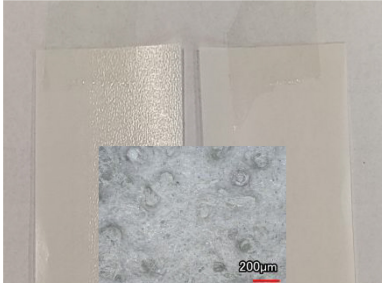
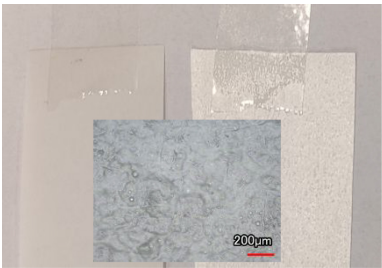
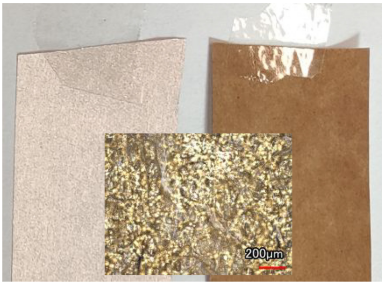
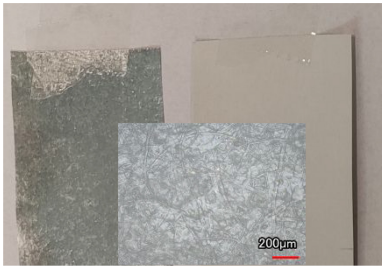
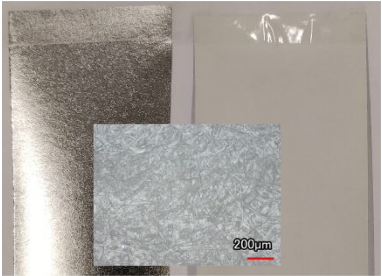
Two commercial Kraft papers were utilized as substrates: one weighing $100\text{g}/\text{m}^2$ for II.a and the other $40\text{g}/\text{m}^2$ for IV.a. The coating process was performed using a Sumet coater (Sumet Technologies GmbH & Co, Germany), employing 40° and 25° angle blades for II.a and IV.a, respectively, at a uniform speed of 5 m/min. The blade pressure was maintained at 10 N, whereas the cylinder pressures were set at 500 N for II.a and 150 N for IV.a. Post-coating, the papers were dried at 80°C using infrared irradiators at 60% intensity for 3 min for II.a and 4 min for IV.a.

2.2.2 | Material Characterization

The thickness measurements of the paper were conducted with a precision thickness gauging model 2010 U (Wolf Messtechnik GmbH, Germany) using a circular shape of 2cm^2 and a pressure of 100 kPa, according to ISO 534 [8]. Twenty readings were taken for each sample. Subsequently, the average thicknesses and standard deviations were calculated. The grammage of paper was determined in accordance with PN-EN ISO 536:2020-08 [9]. For each paper type under examination, six 100cm^2 samples were prepared. These samples were first weighed using a PS 1000.R2 precision balance (Radwag, Poland) to calculate their initial basis weight. Subsequently, they were conditioned for 24 h at 23°C and 50% relative humidity in a climate chamber. Post-conditioning, the samples were re-weighed to determine the final grammage. Average values and standard deviations were calculated. The surface aspect of the coated papers was imaged through optical microscopy (Keyence, Belgium).

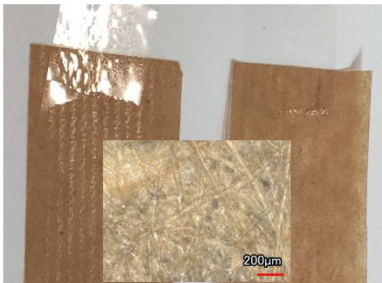
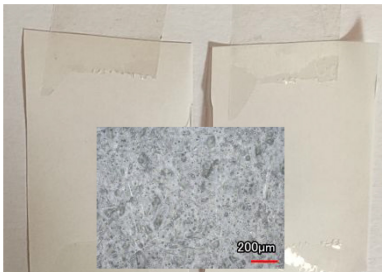
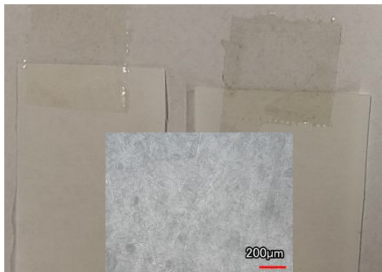
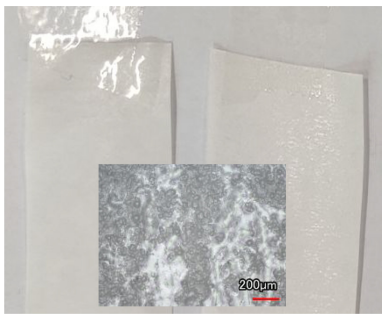
Differential scanning calorimetry (DSC) analysis was done on a DSC 3+ apparatus (Mettler Toledo, Belgium) to determine a glass transition temperature (T_g) and/or melting temperature (T_m) of the coated papers. A single cut disc of coated paper (approx.

TABLE 1 | Code, thickness, grammage, description main component at seal side and appearance of coated papers.

Code Thickness Grammage Description	Images (left: front view; right: seal coating; foreground: microscopic image of the seal coating surface)	Code Thickness Grammage Description	Images (left: front view; right: seal coating; foreground: microscopic image of the seal coating surface)
I.a 65 ± 1 μm 67 ± 0 g/m ² Ethylene, metacrylic acid, acrylate copolymer dispersion		I.b 72 ± 1 μm 45 ± 0 g/m ² Acrylic, polyethylene vinyl acetate copolymer dispersion	
I.c 99 ± 1 μm 102 ± 2 g/m ² Acrylic acid copolymer dispersion		I.d 70 ± 1 μm 69 ± 0 g/m ² Acrylic copolymer dispersion	
I.e 86 ± 1 μm 80 ± 1 g/m ² Proprietary polymeric component dispersion		II.a 146 ± 3 μm 103 ± 1 g/m ² Cellulose nanocrystals dispersion	
II.b 82 ± 2 μm 71 ± 1 g/m ² Vacuum metalized dispersion		II.c 60 ± 1 μm 55 ± 1 g/m ² Vacuum metalized dispersion	
II.d 56 ± 1 μm 67 ± 1 g/m ² Vacuum metalized dispersion		III.a 92 ± 2 μm 71 ± 1 g/m ² Proprietary vegetable wax	

(Continues)

TABLE 1 | (Continued)

Code Thickness Grammage Description	Images (left: front view; right: seal coating; foreground: microscopic image of the seal coating surface)	Code Thickness Grammage Description	Images (left: front view; right: seal coating; foreground: microscopic image of the seal coating surface)
III.b $73 \pm 2 \mu\text{m}$ $44 \pm 1 \text{ g/m}^2$ Ethylene copolymer and wax		IV.a $73 \pm 3 \mu\text{m}$ $41 \pm 2 \text{ g/m}^2$ Polyvinylalcohol (PVOH) dispersion	
IV.b $55 \pm 1 \mu\text{m}$ $45 \pm 0 \text{ g/m}^2$ PVOH dispersion		IV.c $75 \pm 1 \mu\text{m}$ $65 \pm 1 \text{ g/m}^2$ Polyolefin dispersion	
IV.d $97 \pm 1 \mu\text{m}$ $98 \pm 1 \text{ g/m}^2$ Polyethylene (PE) and ethylene vinyl alcohol extrusion		IV.e $126 \pm 3 \mu\text{m}$ $100 \pm 1 \text{ g/m}^2$ Polyolefin extrusion	

20mg) was placed into a sealed aluminium sampling pan and heated between temperatures of -50°C to 150°C (corresponding to the range of heat-seal temperatures), although two subsequent runs at $10^\circ\text{C}/\text{min}$ were recorded. The values for T_g or T_m in the respective transition regions were automatically determined using the STARE Evaluation Software Version 17.00 (Mettler-Toledo, Belgium). The test results of T_g from second heating run are reported, as the first run shows a broad endotherm in the range of 0 – 100°C owing to the presence of water in the paper substrate.

For validation of the coating composition, attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR) characterization was performed in a PerkinElmer Frontier spectrometer with a Golden Gate ATR inset. The coatings were identified using the Spectrum 10 (PerkinElmer, Germany) and KnowItAll (Wiley, USA) software.

2.2.3 | Seal Characterization

Hot tack strengths were measured, following ASTM F1921 [10], using the J&B hot tack tester model 5000 (Vived

Management, Belgium) on 15mm wide strips, sealed with NIPTEF coated jaws of 5 mm seal length. Four fixed seal settings were employed to evaluate the impact of jaw temperature: (i) a seal time of 0.3 s, seal pressure of 0.2 N/mm^2 and a cool time of 0.1 s; (ii) a seal time of 0.3 s, seal pressure of 2 N/mm^2 and a cool time of 0.1 s; a seal time of 1.0 s, (iii) seal pressure of 2 N/mm^2 and a cool time of 0.1 s; and (iv) finally, a seal time of 0.3 s, seal pressure of 2 N/mm^2 and a cool time of 1.0 s. These settings were selected to maintain realistic seal times with both low and high values suitable for the intended flexible packaging concept, vary the seal pressure from low to the device's maximum and alter the cool time from a minimal to a slightly higher yet realistic value. These variations allow subsequent analysis of each setting's influence on the hot tack strength across different jaw temperatures. The jaw temperature increases in increments of 10°C , from initiation to a usual maximum of 180°C , with 5°C steps in critical areas of the hot tack curve, such as during seal initiation and around the peak value. Strength is measured at a grip separation speed of 200 mm/s with a 45 N load cell, consistent with the speed recommendations found in literature for polyolefin seal films [11]. All measurements were performed in triplicate. Average

and standard deviations of maximum strengths, expressed in N/mm, were reported, along with seal failure mechanisms.

Prior to determining cooled seal strengths according to ASTM F88 [12], 30 mm wide strips were sealed using an RDM HSE-3 heat sealer (Benelux Scientific, Belgium) with 25 mm long seal jaws covered in protective Teflon tape. A single fixed seal setting was applied: a seal time of 0.3 s and a seal pressure of 2 N/mm². This setting allows comparison with the similar hot tack setting, albeit with different cool times and considerably slower grip separation speeds, specifically at 300 mm/min, in accordance with the standard, characteristic of these experiments. The jaw temperature increased in increments of 20°C, starting from initiation to a typical maximum of 180°C, with increments of 5°C during seal initiation. After sealing, 15 mm strips were cut from the centre using a sample cutter. Following a 4-h cool period, these thin strips were tested using a Tinius Olsen 5ST universal testing machine equipped with stainless steel clamps and diamond-coated jaws (Benelux Scientific, Belgium). Strengths were measured using a 250 N load cell at a grip separation speed of 300 mm/min. All measurements were performed in triplicate. Average and standard deviations of maximum strengths, expressed in N/mm, were reported, along with seal failure mechanisms.

To evaluate the thermal resistance of coated papers under various seal settings, corresponding to those used in experiments to determine hot tack and seal strengths, interface temperatures were measured using a Type K precision membrane thermocouple (TC Direct, Netherlands). These thermocouples, 0.13 mm thick and 12 × 20 mm in size, were pre-embedded between two polyamide films and cured, offering response times in the millisecond range. Data acquisition was performed using Omegalog v3.9 software (Omega Engineering, USA), with one measurement each 45 ms. In Figure 1, the attachment of the coated paper is shown, centrally positioned within a cardboard tool (left), which features an opening sufficiently large to allow unimpeded opening and closing of the seal jaws of the RDM sealer. On the right, it is evident that the membrane thermocouple is situated between two layers of coated paper. This set-up simulates the seal process, enabling temperature

measurement at the precise location of actual sealing, specifically at the seal coating. For each type of paper tested, five repetitions are conducted per seal setting, with the average and standard deviation calculated for the measured maximum temperatures. Measurements were also conducted without coated paper, using the bare membrane thermocouple, to subsequently determine the net thermal resistance of coated papers by eliminating the additional influence of time and pressure on the thermal resistance of the membrane itself surrounding the thermocouple.

3 | Results

3.1 | Material Characterization

Table 1 provides images, surface aspect, measured thicknesses and grammages of coated papers. These papers are categorized into four classes, designated as classes I through IV based on initial coating composition knowledge as validated with ATR-FTIR. Each class shows distinct thermal behavioural differences as confirmed by DSC. The papers within each class are further numbered using Roman numerals and alphabets (e.g. I.a, I.b, II.a) for clarity. The allocation and detailed discussion of these classes will be presented in the subsequent section.

3.1.1 | DSC

The DSC curves recorded on coated papers are illustrated in Figure 2. Despite a relatively small coating layer present, good sensitivity of the characterization technique is experienced showing distinct thermal properties depending on the coating type for all samples. The thermogram during second heating is plotted for determination of glass transition temperature (T_g) and/or melting temperature (T_m), as the first heating step is highly influenced by a broad endothermal peak representing evaporation of equilibrium water content in the paper substrate. Based on the DSC results and depending on thermal characteristics, the coated papers can be assigned to the

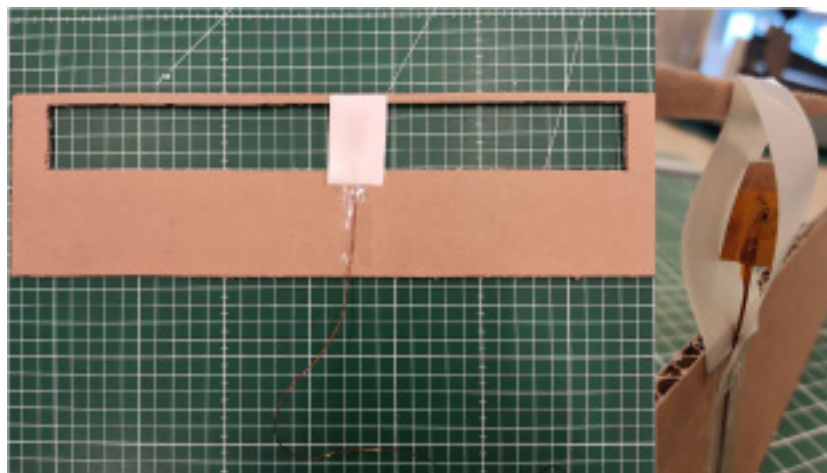


FIGURE 1 | Left: Coated paper centred on cardboard tool for interface temperature measurement (°C). Right: Membrane thermocouple at coated paper seal interface.

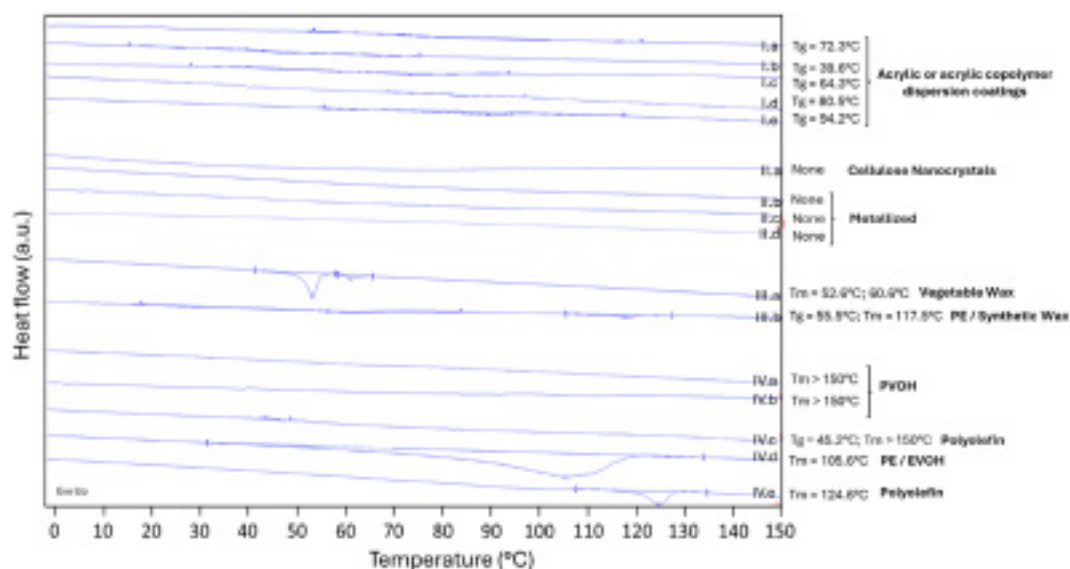


FIGURE 2 | Thermal behaviour of coated papers as represented by thermograms recorded by DSC measurements for different coating types, with indication of glass transition temperatures (T_g) and/or melting temperatures (T_m).

different classes as presented. The polymer dispersion coatings are typically characterized by a T_g as expected, for example, for acrylic or acrylic copolymer (e.g. acrylic/styrene- or acrylic/vinyl-based) polymer coatings depending on the commercial polymer grade. These coatings are identified by distinct T_g and show typical softening effects in the rubbery state allowing for favourable heat-seal performance as further evaluated. Five papers with these coatings are classified as Class I. The coated papers without thermal transitions in the scanned temperature region correspond to inert coatings or inorganic coatings, including metallized papers or paper coatings with cellulose nanocrystals. None of the metallized coatings nor the cellulose nanocrystals do show thermal transitions. Slight transitions may only be attributed to water sorption phenomena in the base paper that cause minor plasticity effects and are most pronounced for the cellulose nanocrystal coatings. Four of these coated papers are categorized as Class II. The presence of wax coatings is clearly detected by a well-determined melting interval of the wax with typically multiple melting peaks corresponding to the composition of the specific wax. The vegetable wax is clearly identified by a lower melting temperature compared to the synthetic wax. Composite coatings of thermoplastics with wax are also characterized by separate melting points corresponding to each of the constituents. Two papers with either vegetable or synthetic wax-based coatings are denoted as Class III. The other types of thermoplastic (laminated) coatings show a clear melting regime at expected melting intervals of polyolefins [e.g. polyethylene (PE)] or with melting temperatures beyond the temperature limits of present scanning range, which was limited to 150°C in parallel with the practical settings of the heat-sealing tests [e.g. polyvinyl-alcohol (PVOH), ethylene-vinyl alcohol (EVOH)]. Five papers with these coatings are identified as Class IV. It is interesting to observe that the respective softening and melting intervals of the coatings can be clearly determined in contrast with the thermal inertness of the paper substrate in present temperature region, which will further be related to the selection of appropriate heat-seal temperatures.

3.2 | Seal Characterization

3.2.1 | Hot Tack Strength

For each of the 16 coated papers, four hot tack curves are available, corresponding to different settings of seal time, seal pressure and cool time as shown in Figure 3. Due to the extensive data volume, select characteristics of these curves are extracted to show the influences of seal time, seal pressure and cool time on each coated paper. These characteristics, also illustrated in the figure for the red coloured setting, are defined as follows: (i) The peak value represents the maximum strength achieved, for which the average and standard deviation are provided; (ii) the seal window is the temperature range where the strength exceeds 0.1 N/mm by the average plus one standard deviation; (iii) the seal initiation temperature is the minimum temperature at which a threshold of 0.03 N/mm is surpassed by the mean plus one standard deviation [13].

The three seal characteristics are subsequently used in Table 2, along with a description of the seal failure mechanisms, for all coated papers to illustrate the impact of the seal settings on hot tack performance.

Generally, a clear relationship emerges between hot tack strength and cohesive and fibre tear failures. Samples with adhesive failure have a very low strength. The two internally coated papers (i.e. samples II.a and IV.a) have negligible to very weak hot tack strength.

Seal time, at the explored values of 0.3–1.0 s, mostly has limited to no impact on hot tack strength, seal initiation temperature and the seal window, likely due to the low grammages and thicknesses of the papers considered in this study, which facilitates rapid heat transfer to the seal interface. Impact of seal time is most pronounced, albeit modestly, in sample II.b, where sealing initiates 10°C earlier at a high seal time of 1.0 s. A moderate hot tack strength around 0.10–0.13 N/mm is maintained at this seal

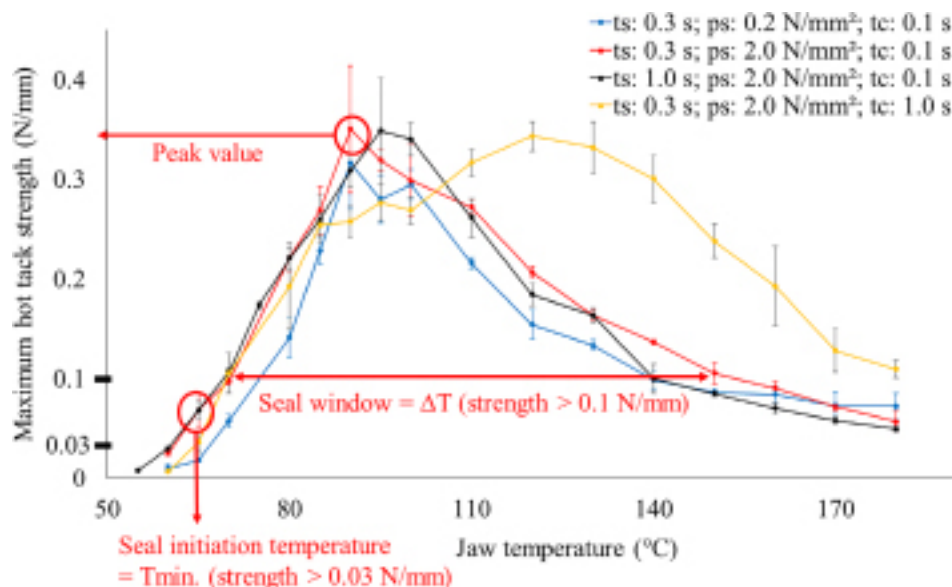


FIGURE 3 | Illustrative curves for hot tack strength values of various seal time (ts), pressure (ps) and cool time (tc) settings of sample I.a, highlighting peak value, seal window and seal initiation temperature of the red-coloured seal setting (ts: 0.3 s, ps: 2.0 N/mm², tc: 0.1 s).

time, although the curve for seal time of 0.1 s largely remains below the 0.10 N/mm threshold. Beyond just reaching a temperature suitable for sealing, for some papers with coatings that are not as evenly distributed, a longer seal time might enable the seal polymer to flow over the surface and then smooth out the coating, when the seal jaws are closed. This process could lead to a better distribution of entanglements across the seal surface, potentially increasing strength.

Conversely, seal pressure, at the explored values of 0.2 and 2.0 N/mm², considerably influences hot tack characteristics in many coated papers, with an exceptional positive effect observed in increasing seal pressure for samples I.d, III.a, IV.a and IV.c. This is evidenced by increased peak values, reaching an amplification of double or more, lowered initiation temperature and a broader seal window. For these papers, high pressure appears essential for achieving sufficient hot tack performance. Modest improvements are also noted in samples I.a, I.b, I.c, I.e, II.d, IV.b, IV.d and IV.e by increasing seal pressure. Both enhanced thermal conductivity, resulting from the more intense compression of paper fibres at high pressure, which displaces air that acts as a thermal insulator, and a potential additional smoothing effect on the coating, analogous to the previous explanation for the impact of seal time, can improve seal performance, in terms of both lower seal initiation temperatures and higher maximum strengths.

Cool time, at the explored values of 0.1 and 1.0 s, generally enhances hot tack strength through longer cooling. And this is to be expected, given the generally higher strength of the cooled polymer mass, especially if it returns to solid state during the brief cool time. Sample III.b is the most notable, with almost no measurable strength without sufficient cooling. Other papers (e.g. samples IV.b, IV.c and IV.d) double in strength after 1.0 s of cool time, also considerably widening the seal window. However, for paper samples I.d, II.c, IV.a and IV.d, the combination of enhancement in hot tack strength and seal window expansion is more modest. Finally, no measurable hot tack enhancement is observed in samples I.a, I.b, I.e and III.a with 1.0 s of cooling.

When analysing the hot tack strengths according to the paper classes previously defined by DSC, several general observations can be made. Class I papers are typically quite strong. Papers in classes II and III are largely very weak. Class IV papers are also relatively weak but tend to become strong with just a minimal cooling of 1 s. The foundation of these variations in seal performance lies in the different bonding mechanisms, combined with the amorphous or semi-crystalline nature, specific to the polymer type. These aspects will be further discussed later.

3.2.2 | Cooled Seal Strength

Analogous to the hot tack findings, the results of tests measuring seal strengths 4 h post-sample preparation are presented in tabular form. Two numerical key characteristics are highlighted: the peak value, corresponding to a plateau value observable in the seal strength curve for sample I.a in Figure 2, and the seal initiation temperature, for which a slightly higher threshold of 0.05 N/mm is employed compared to hot tack, consistent with a previous methodology in the literature [14] (Figure 4).

These two numerical seal characteristics, along with the failure mechanism, are listed in Table 3.

Generally, it is evident that the majority of coated papers fail via fibre tear after extended cooling. Notably, two of the three papers that do not exhibit this mechanism (i.e. samples II.a and II.b), both belonging to Class II, and have very low seal strengths, with maximum values well below 0.1 N/mm. Previously, the hot tack results in Table 2 already highlighted the low strengths of these papers. At maximum strength, a 15 mm seal would already open under a minimal force of less than 1 N. Therefore, even in the cooled state, these very weak seal strengths, rendering them unsuitable for withstanding subsequent processes. This includes resistance to stresses encountered during transportation, storage and handling in

TABLE 2 | Influence of seal time, seal pressure and cool time on hot tack characteristics (peak value, seal initiation temperature, seal window and failure mechanism) of 16 coated papers. ‘/’ signifies the characteristic is indeterminable, as the threshold value remains unexceeded.

Code	Hot tack characteristic	Seal time (s)	Seal pressure (N/mm ²)	Cool time (s)
		0.3→1.0	0.2→2.0	0.1→1.0
I.a	Peak value (N/mm)	0.35 ± 0.06 → 0.35 ± 0.06	0.32 ± 0.05 → 0.35 ± 0.06	0.35 ± 0.06 → 0.34 ± 0.01
	Seal initiation temperature (°C)	65 → 60	70 → 65	65 → 65
	Seal window (°C)	80 → 70	60 → 80	80 → 110
	Seal failure mechanism	Mainly cohesive seal failure at low cool time, while at high cool time, fibre tear becomes the dominant mechanism		
I.b	Peak value (N/mm)	0.28 ± 0.01 → 0.27 ± 0.02	0.26 ± 0.03 → 0.28 ± 0.01	0.28 ± 0.01 → 0.25 ± 0.02
	Seal initiation temperature (°C)	65 → 65	70 → 65	65 → 70
	Seal window (°C)	90 → 85	110 → 90	90 → 100
	Seal failure mechanism	Mainly cohesive seal failure at low cool time, while at high cool time, fibre tear becomes the dominant mechanism		
I.c	Peak value (N/mm)	0.52 ± 0.03 → 0.54 ± 0.00	0.39 ± 0.02 → 0.52 ± 0.03	0.52 ± 0.03 → 0.55 ± 0.05
	Seal initiation temperature (°C)	60 → 55	65 → 60	60 → 60
	Seal window (°C)	90 → 100	110 → 90	90 → 100
	Seal failure mechanism	Mainly fibre tear		
I.d	Peak value (N/mm)	0.22 ± 0.01 → 0.24 ± 0.01	0.10 ± 0.01 → 0.22 ± 0.01	0.22 ± 0.01 → 0.33 ± 0.02
	Seal initiation temperature (°C)	85 → 80	110 → 85	85 → 80
	Seal window (°C)	70 → 100	/ → 70	70 → 85
	Seal failure mechanism	Adhesive seal failure at low cool time and low seal pressure, mainly cohesive seal failure at high seal pressure and fibre tear at high cool time		
I.e	Peak value (N/mm)	0.63 ± 0.06 → 0.58 ± 0.05	0.59 ± 0.04 → 0.63 ± 0.06	0.63 ± 0.06 → 0.44 ± 0.05
	Seal initiation temperature (°C)	90 → 85	90 → 90	90 → 95
	Seal window (°C)	90 → 90	80 → 90	90 → 80
	Seal failure mechanism	Mainly fibre tear		
II.a	Peak value (N/mm)	0.02 ± 0.00 → 0.01 ± 0.00	0.01 ± 0.00 → 0.02 ± 0.00	0.02 ± 0.00 → 0.03 ± 0.00
	Seal initiation temperature (°C)	/	/	/
	Seal window (°C)	/	/	/
	Seal failure mechanism	Adhesive seal failure		
II.b	Peak value (N/mm)	0.11 ± 0.01 → 0.13 ± 0.00	0.10 ± 0.00 → 0.11 ± 0.01	0.11 ± 0.01 → 0.13 ± 0.01
	Seal initiation temperature (°C)	85 → 75	90 → 85	85 → 80
	Seal window (°C)	/ → 90	/	/ → 90
	Seal failure mechanism	Mainly cohesive seal failure		
II.c	Peak value (N/mm)	0.13 ± 0.04 → 0.13 ± 0.01	0.15 ± 0.01 → 0.13 ± 0.04	0.13 ± 0.04 → 0.16 ± 0.02
	Seal initiation temperature (°C)	75 → 70	80 → 75	75 → 75
	Seal window (°C)	45 → 70	90 → 45	45 → 70
	Seal failure mechanism	Mainly cohesive seal failure		

(Continues)

TABLE 2 | (Continued)

Code	Hot tack characteristic	Seal time (s)	Seal pressure (N/mm ²)	Cool time (s)
		0.3→1.0	0.2→2.0	0.1→1.0
II.d	Peak value (N/mm)	0.25 ± 0.01 → 0.27 ± 0.01	0.21 ± 0.01 → 0.25 ± 0.01	0.25 ± 0.01 → 0.26 ± 0.02
	Seal initiation temperature (°C)	75 → 70	80 → 75	75 → 70
	Seal window (°C)	90 → 80	70 → 90	90 → 105
	Seal failure mechanism	Mainly cohesive seal failure at low cool time, while at high cool time, fibre tear becomes the dominant mechanism		
III.a	Peak value (N/mm)	0.14 ± 0.00 → 0.14 ± 0.00	0.05 ± 0.01 → 0.14 ± 0.00	0.14 ± 0.00 → 0.13 ± 0.00
	Seal initiation temperature (°C)	115 → 120	180 → 115	115 → 130
	Seal window (°C)	70 → 120	/ → 70	70 → 40
	Seal failure mechanism	Mainly cohesive seal failure at low cool time, while at high cool time, fibre tear becomes the dominant mechanism		
III.b	Peak value (N/mm)	0.01 ± 0.00 → 0.01 ± 0.00	0.01 ± 0.00 → 0.01 ± 0.00	0.01 ± 0.00 → 0.05 ± 0.01
	Seal initiation temperature (°C)	/	/	/ → 100
	Seal window (°C)	/	/	/
	Seal failure mechanism	Adhesive seal failure		
IV.a	Peak value (N/mm)	0.07 ± 0.01 → 0.08 ± 0.01	0.01 ± 0.00 → 0.07 ± 0.01	0.07 ± 0.01 → 0.17 ± 0.06
	Seal initiation temperature (°C)	100 → 95	/ → 100	100 → 90
	Seal window (°C)	/	/	/ → 30
	Seal failure mechanism	Adhesive seal failure		
IV.b	Peak value (N/mm)	0.09 ± 0.01 → 0.11 ± 0.01	0.09 ± 0.01 → 0.09 ± 0.01	0.09 ± 0.01 → 0.21 ± 0.00
	Seal initiation temperature (°C)	85 → 85	90 → 85	85 → 90
	Seal window (°C)	/	/	/ → 20
	Seal failure mechanism	Adhesive seal failure at low cool time, while at high cool time, fibre tear becomes the dominant mechanism		
IV.c	Peak value (N/mm)	0.11 ± 0.00 → 0.13 ± 0.01	0.02 ± 0.01 → 0.11 ± 0.00	0.11 ± 0.00 → 0.24 ± 0.00
	Seal initiation temperature (°C)	90 → 85	/ → 90	90 → 90
	Seal window (°C)	/	/ → /	/ → 20
	Seal failure mechanism	Adhesive seal failure at low cool time and low seal pressure, mainly cohesive seal failure at high seal pressure and fibre tear at high cool time		
IV.d	Peak value (N/mm)	0.28 ± 0.01 → 0.21 ± 0.01	0.23 ± 0.01 → 0.28 ± 0.01	0.28 ± 0.01 → 0.37 ± 0.02
	Seal initiation temperature (°C)	95 → 90	95 → 95	95 → 90
	Seal window (°C)	65 → 70	40 → 65	65 → 65
	Seal failure mechanism	Mainly cohesive seal failure at low cool time, while at high cool time, fibre tear becomes the dominant mechanism		
IV.e	Peak value (N/mm)	0.10 ± 0.01 → 0.11 ± 0.00	0.07 ± 0.00 → 0.10 ± 0.01	0.21 ± 0.01 → 0.37 ± 0.02
	Seal initiation temperature (°C)	110 → 110	120 → 110	110 → 105
	Seal window (°C)	/	/	/ → 40
	Seal failure mechanism	Adhesive seal failure at low cool time and low seal pressure, mainly cohesive seal failure at high seal pressure and fibre tear at high cool time		

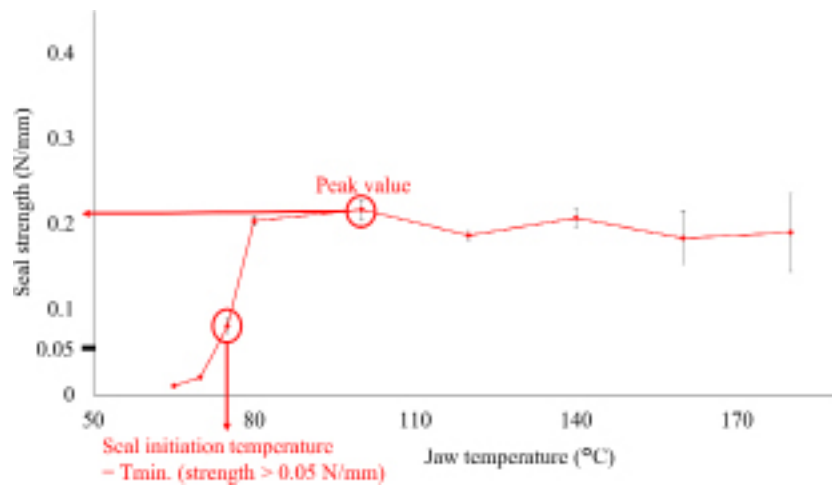


FIGURE 4 | Illustrative curve for cooled seal strength values of sample I.a, highlighting peak value, and seal initiation temperature, sealed at 0.3 s seal time and 2.0 N/mm² seal pressure.

TABLE 3 | Seal strength characteristics (peak value, seal initiation temperature and failure mechanism) of 16 coated papers, sealed at a seal time of 0.3 s and a seal pressure of 2.0 N/mm².

Code	Seal strength characteristic		
	Peak value (N/mm)	Seal initiation temperature (°C)	Seal failure mechanism
I.a	0.22 ± 0.01	75	Mainly fibre tear
I.b	0.19 ± 0.02	75	Mainly fibre tear
I.c	0.56 ± 0.05	70	Mainly fibre tear
I.d	0.29 ± 0.01	90	Mainly fibre tear
I.e	0.69 ± 0.04	120	Mainly fibre tear
II.a	0.06 ± 0.01	185	Adhesive peel
II.b	0.06 ± 0.01	120	Mainly cohesive peel
II.c	0.15 ± 0.01	80	Mainly cohesive peel
II.d	0.27 ± 0.01	75	Mainly fibre tear
III.a	0.24 ± 0.01	130	Mainly fibre tear
III.b	0.29 ± 0.02	75	Mainly fibre tear
IV.a	0.16 ± 0.05	100	Mainly fibre tear
IV.b	0.28 ± 0.02	105	Mainly fibre tear
IV.c	0.28 ± 0.04	100	Mainly fibre tear
IV.d	0.74 ± 0.03	100	Mainly fibre tear
IV.e	1.14 ± 0.06	125	Mainly fibre tear

retail environments, such as vibrations, shocks and sustained forces. For papers from classes I, III and IV, no clear trend is evident.

Hot tack and cooled seal strengths are distinct properties, involving different sealers and varying grip separation speeds, with hot tack resembling an impact test. To facilitate a prudent comparison, efforts were made to standardize settings as much as possible, such as maintaining constant seal time at

0.3 s and seal pressure at 2.0 N/mm². However, comparisons should still be approached with caution. It is of great practical importance to consider both strengths in evaluating seal quality, as they are crucial immediately after jaw opening and during subsequent steps, with temperature data being vital for accurate seal jaw settings. In this context, comparing peak values and seal initiation temperatures from both experiments, under these controlled conditions, is highly informative. A considerable number of the 14 other coated papers

potentially suitable for primary food applications show strong similarities in initiation temperatures. It is advisable to set the seal jaws at a temperature higher than the initiation value, considering the subsequent increase in strength with temperature and potential deviations in the sealer's temperature, yet low enough to remain within the seal window. For paper samples I.a, I.b, I.c, I.d, I.e, II.d, III.a and IV.d, this can be safely achieved without cooling due to their temperature alignment and a relatively broad seal window. The seal coatings of these papers remain quite strong in a rubbery or molten state. A few coated papers, such as samples IV.b, IV.c, IV.e and III.b, may be considered for safe heat-sealable primary food packaging if adequately cooled immediately after opening the jaws and/or used with lightweight food items. The seal coatings of these papers are relatively weak in a rubbery or molten state, necessitating cooling to strengthen the seal. Finally, aside from the previously mentioned unsuitable papers, there are some borderline cases with relatively weak seal strengths (e.g. samples II.c and IV.a), where extra caution and potentially preliminary enhancements to the paper to improve seal performance might be necessary during potential implementation. It is possible that the coatings of these papers are poorly distributed across the seal surface and/or are present in limited amounts, resulting in weaker mechanical strengths after sealing, compared to the other papers.

Beyond the data presented in the tables, two additional observations are noteworthy to be further detailed and to better understand the sealing quality. The first observation is that several coated papers are unsuitable for sealing at very high temperatures due to air pocket formation within the paper. These air pockets can partially and, in some cases almost completely, open the seal due to the pressure exerted by one or more air pockets. This phenomenon starts to occur at seal jaw temperatures of 160°C in samples II.b, II.c, IV.d and IV.e, and only at 180°C in samples I.d, IV.a, IV.c and I.c. Given the decreased hot tack

strengths at these temperatures, it is already inadvisable to heat these papers excessively; however, air pocket formation provides an additional reason to avoid excessive temperatures. The most extreme example of such air pockets in seals of sample II.c is illustrated in Figure 3. Air pocket formation can be explained by (i) the expansion of air already present in the paper at high temperatures and/or the displacement of some of the air in the paper towards the seal interface and (ii) the insufficient removal of air at the seal interface due to the encapsulation of the air bubble by entanglements that occur between the seal polymers on either side. The second observation is a noticeable decrease in the seal plateau for several coated papers. Though largely attributable to air pockets, an exceptional case where this is not the cause is sample III.b. The seal strength curve for sample III.b, shown in Figure 3, clearly demonstrates a near-total reduction to 0 N/mm at 180°C, without the formation of air pockets (Figure 5).

3.2.3 | Seal Interface Temperature

To gain better understanding of how seal time and pressure influence hot tack performance in certain papers, interface temperatures were measured. This was to determine if these effects stemmed from the paper's thermal resistance. Tests focused on sample II.b, most sensitive to seal time, and sample I.d, most responsive to seal pressure. Additionally, comparisons were made between a high grammage paper (e.g. sample II.a) and a low grammage paper (e.g. sample IV.b).

In Figure 6, the interface temperature curves for each type of paper and for the bare membrane thermocouple are displayed. The bare thermocouple closely approaches the set temperature of 120°C, reaching a maximum of approximately 114°C with a 0.3 s seal jaw closure. The maximum temperature with coated paper is substantially lower due to thermal resistance. The paper

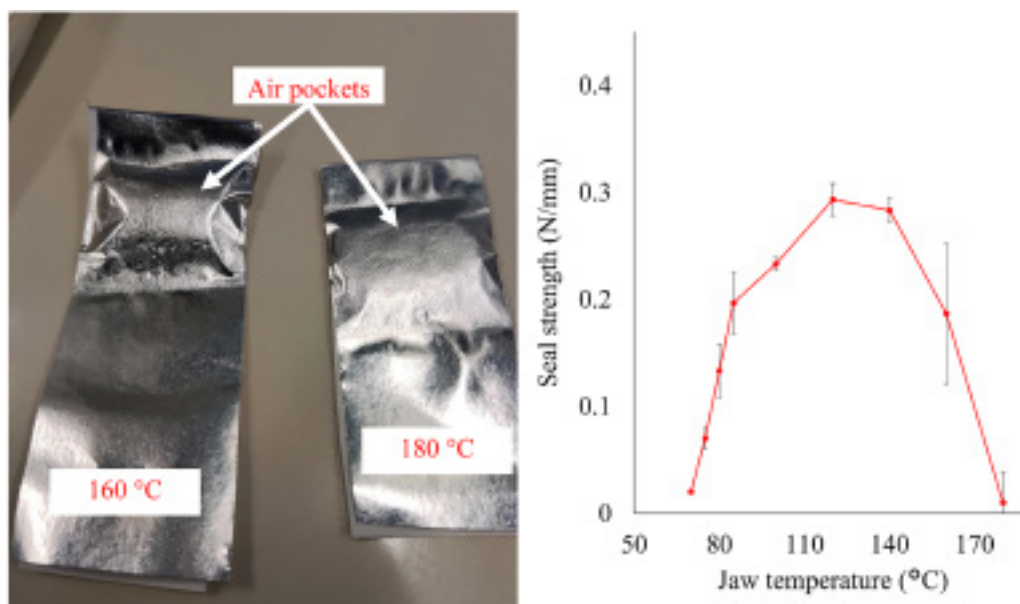


FIGURE 5 | Additional observations after sealing: air pockets in the seal area of sample II.c after sealing at 160 and 180°C (left). Seal strength curve of sample III.b, sealed at 0.3 s seal time and 2.0 N/mm² seal pressure, at jaw temperatures of 160°C and 180°C, a noticeable reduction in seal strength is observed (right).

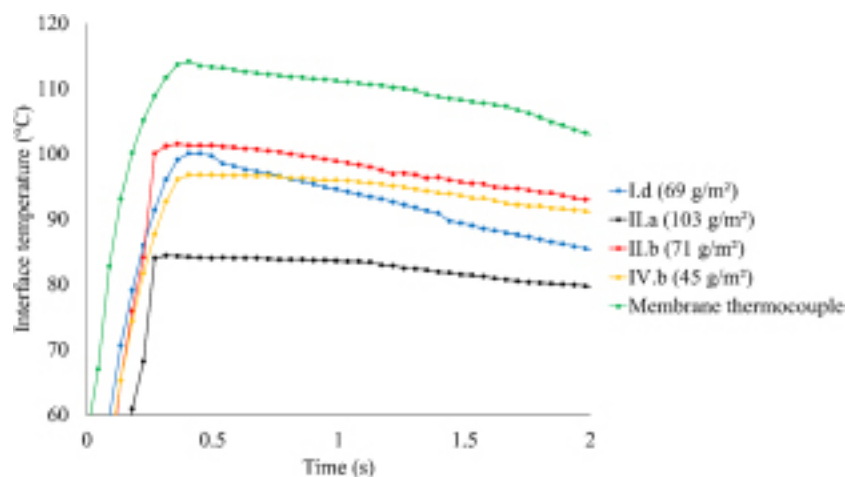


FIGURE 6 | Comparison of interface temperature curves for coated papers via membrane thermocouple: with and without paper (solely membrane thermocouple) at a seal setting of 120°C, 0.3 s and 2.0 N/mm².

TABLE 4 | Average values and standard deviations of maximum interface temperature for varied seal times and pressures at fixed 120°C jaw temperature ($n = 5$).

Material code	Grammage (g/m ²)	Seal conditions			
		Seal time (s)	0.3		1.0
			0.2	2.0	2.0
I.d	69		80.2 ± 1.2	99.8 ± 1.9	117.8 ± 0.2
II.a	103		63.8 ± 1.3	84.0 ± 1.2	114.1 ± 0.4
II.b	71		84.5 ± 1.0	100.4 ± 0.8	117.3 ± 0.1
IV.b	45		77.5 ± 1.7	96.0 ± 1.8	117.3 ± 0.3
Membrane thermocouple			101.6 ± 0.8	113.7 ± 0.9	120.0 ± 0.1

with the highest grammage of 103 g/m² achieves the lowest maximum, around 84°C. For other papers, maximum temperatures are close, ranging between 97°C and 102°C. Notably, the paper with the lowest grammage of 45 g/m² shows higher thermal resistance than the other two heavier papers. This is also evident from results at a high seal time of 1.0 s and low seal pressure of 0.2 N/mm², as shown in Table 4. The cool rates of the curves in Figure 6 are also of interest. It is clear that the heavier paper cools down slower than the lighter ones. Previous results from the hot tack strength tests indicate that insufficiently rapid cooling can negatively affect strength immediately after opening the seal jaws.

Table 4 results reveal the influence of seal time and pressure on the maximum interface temperature. Without coated paper, with only a bare membrane thermocouple, it is clear that higher seal times increase the maximum interface temperature, as does increasing the seal pressure. At a seal time of 1.0 s and pressure of 2.0 N/mm², the interface temperature reaches the set temperature of the jaws, namely, 120°C. The maximum interface temperatures with coated papers also approach the set temperature under these conditions, with very low standard deviations observed. At a short seal time of 0.3 s, with both low and high

seal pressures of 0.2 and 2.0 N/mm², standard deviations are slightly higher, up to about 2°C. This variation could be due to the sealer's error at such a low seal time or due to the method's error, as a result of the limited time causing greater variations in the positioning of the fluctuating thermocouple between the seal jaws. The latter is a less expected issue at longer seal times, where optimal contact between the thermocouple, surrounding materials and seal jaws is expected.

By subtracting the temperature readings of the coated papers from those of the bare membrane thermocouple at higher temperatures, the thermal resistance is quantified. It is expressed in degrees Celsius, indicating the extent to which the interface temperature is lower than that of the bare thermocouple, attributable to the presence of the coated paper. There is a limited correlation between these values and the thickness and grammages of the papers (not shown). As expected, thermal resistance is much higher in thicker and heavier paper. Interestingly, as also shown in Figure 6, among the three lower grammage papers, which are also thinner, the lowest grammage paper (sample IV.b), which is also the thinnest, shows slightly higher thermal resistance than the other two papers with basis weights of 69 and 71 g/m² (samples II.a and II.b).

4 | Discussion

4.1 | Influence of Seal Parameters on Seal Performance

This study's findings reveal a considerable positive impact of cool time, seal pressure and, to a lesser extent, seal time on hot tack strength, a trend consistent with previous literature sources as below. Such influences on the hot tack strength of coated paper were initially identified in a seal study of extrusion coated paper [15]. These results are further supported by a more recent study, examining the seal performance of paper-based packaging, which varied seal time and pressure and observed hot tack strength. The observed increase in strength was attributed to improved heat transfer [16]. Another recent study in this field observed that the effect of seal pressure on seal strength becomes negligible beyond a threshold of 3 bar, suggesting a requisite pressure level for optimal seal strength. Notably, this study focused on cooled seal strength, which is presumably less sensitive to minor variations in seal parameters than the hot tack method [17]. In addition to these parameters, the impact of jaw temperature is critical in heat conductive sealing, as insufficient heat supply renders other parameters insignificant in influencing strength. The observed influences of jaw temperature on hot tack and seal strength align with findings from the previous studies [10–12, 18]. The hot tack curve shows a distinct peak strength, decreasing at higher temperatures. The seal strength curve has a sigmoidal pattern, reaching a plateau strength upon surpassing a threshold value. This sigmoidal shape is attributed to the failure mechanism where the paper substrate exhibits fibre tear and/or cohesive failure occurs, preventing further increases in strength. This plateau value eventually decreases, potentially due to air pocket formation causing partial seal opening, or other factors, as observed in the wax-coated paper (e.g. sample III.b). In this case, the seal coating may be expelled or absorbed by the paper at higher jaw temperatures.

4.2 | Polymer Composition and Seal Performance Dynamics

The differences in seal performance between Class I and Class IV papers can be attributed to their distinct coating compositions, predominantly acrylic copolymers in the former and polyolefins and PVOH in the latter. Notably, a trend is observed where Class I papers exhibit a relatively strong hot tack strength immediately after 0.1 s cool time and gain little additional strength with further cooling. In contrast, Class IV papers start with relatively low strength after 0.1 s but show a marked increase in strength with extended cooling durations. This can be related to the forces occurring within the polymer mass shortly after sealing. Initially, however, polymer chains must diffuse during the sealing process and subsequently entangle with each other [19]. Chain mobility is of crucial importance. In largely amorphous polymers like acrylic copolymers, the T_g needs to be exceeded, whereas in semi-crystalline polymers like PE, the crystal structure must be largely melted for participation of long chains in the diffusion process [2]. After entanglement, materials achieve stronger hot tack either through high melt strength, rapid crystallization of chains within the molten mass or a combination of both, varying with the type of polymer involved [5]. This strength is attained by adequate cooling and/or additional chemical bonds that maintain the

integrity of the melt. Acrylic copolymers, besides chain entanglement from diffusion, are further held together by hydrogen bonding. In contrast, polyolefins have weaker melt strength due to a lack of additional bonds like hydrogen bonds. Its melt strength relies on cooling and partial recrystallization of the molten polymer [5, 20]. This is notably illustrated when comparing the relatively weaker results of polyolefin seals after 0.1 s cool time with their considerably stronger outcomes following a 1.0 s cooling period. Similarly, in another identified semi-crystalline polymer in Class IV papers, PVOH found in samples IV.a and b, cooling is also crucial for strong hot tack. Although PVOH can form hydrogen bonds, like acrylic copolymers, hydrogen bonding appears to play a less notable role in the melt strength development. Similar to polyolefin coated papers in Class IV, coated papers with PVOH are characterized by narrow hot tack windows and low strengths. Class II coated paper seals, with apparent thermal inertia in DSC, have very low hot tack and cooled seal strengths likely due to insufficient thermoplastic presence, as detected by DSC, resulting in the absence of sufficient chain entanglements to increase seal strength substantially. Class III paper seals, similar to Class IV, need adequate cooling. This could be attributed to the nonpolar nature of wax polymers, which results in the chains being predominantly held together by chain diffusion rather than hydrogen bonds or polar or ionic interactions. The hot tack strength of sample III.b is non-measurable, and ethylene-wax blend (e.g. sample III.a) mirrors Class IV papers, with weak but measurable hot tack strength and enhanced post-cooling seal strength. Among the selection of 16 materials, the seal performances of primarily acrylic-based coatings from Class I and polyolefin-based coatings from Class IV are the most prominent.

Possibly related to this, there is a difference in thermal resistance among papers with low grammage. It appears counterintuitive that the lighter, thinner paper IV.b shows higher thermal resistance at a short closing time of 0.3 s, compared to the slightly thicker and heavier papers I.d and II.b. This may be attributed to whether or not energy is consumed in melting the crystals in the polymer coating, where the thickness of the coating also plays a crucial role by providing more material to absorb energy. In this case, the coating of sample IV.a or IV.b will melt, consuming significantly more energy, which is evident in the DSC thermogram. Meanwhile, the coatings of samples I.d and II.b appear more amorphous in the DSC thermogram as represented by a single T_g , indicating different thermal behaviours. Of course, there are other potential causes for the increased thermal resistance, such as the thermal insulation resulting from the paper substrate. Regarding this latter possible cause, due to the nature of the research, which primarily involved working with finished commercial papers, it is challenging to make further assertions.

4.3 | Correlating DSC Transition Temperatures With Seal Initiation

Based on the theoretical principles of sealing, which require exceeding either the T_g or the T_m , depending on the polymer type to allow sufficient mobility of polymer chains to initiate sealing, it is pertinent to compare the transition temperatures identified via DSC with the seal initiation temperatures. These initiation temperatures were determined through stepwise measurement of cooled seal strengths. This comparison is illustrated in

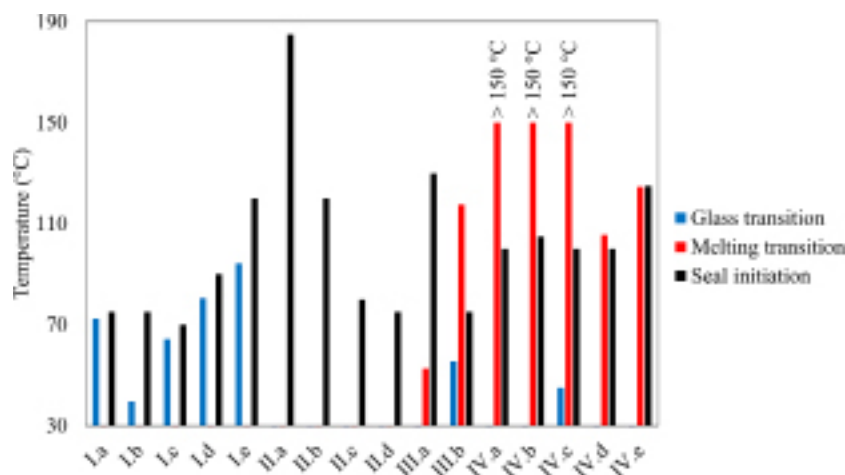


FIGURE 7 | Comparative evaluation of glass transition and melting temperatures (determined by DSC) and seal initiation temperatures (determined by cooled seal strength testing) across 16 coated paper samples.

Figure 7, showing that seal initiation generally occurs at temperatures higher than the relevant transition temperatures of the polymers in the coating. The thermocouple measurements showed that the actual interface temperature is considerably lower than the jaw temperature at 0.3 s seal time. As a result, the T_g values and the actual interface temperature at seal initiation are even closer for the Class I papers. For coated papers that require surpassing a melting temperature T_m , such as those in Class III and Class IV, this method appears to be less sensitive in determining seal initiation temperature through DSC analysis. The accuracy of this method may also be influenced by the coating thickness and, consequently, the quantity of thermoplastic material present. Finally, the paper coating comprises not only seal polymers but also other thermoplastics to provide oil/grease, water, vapour, oxygen gas, aroma or other barrier properties. Despite these nuances, DSC shows potential for offering initial insights into the seal performance of coated paper. It does so by indicating seal initiation temperatures of Class I coated papers. Additionally, it assesses the need to melt crystals, a factor linked to specific seal behaviour observed in Class III and IV papers as described in this study. Furthermore, DSC considers the quantity of thermoplastic material present, which serves as an indicative parameter for effective seal performance.

5 | Conclusion

This study used DSC testing with varied temperatures to highlight transitions that inform seal performance in commercial coated papers. The correlation of these transition temperatures with seal initiation temperatures, although not robust, offers preliminary insights into the conditions necessary for effective sealing. Class I papers, primarily featuring acrylic-based coatings, exhibited strong hot tack, with minimal strength gain upon further cooling, reflecting their amorphous polymer characteristics and likely benefiting from additional bonds such as hydrogen bonding, contributing to the relatively high strength of the molten mass. Class II coated papers exhibit weak hot tack and cooled seal strengths likely due to insufficient thermoplastic material and resultant limited chain entanglements necessary for high strength. Class III papers, with wax-based coatings,

exhibit negligible to weak hot tack strengths, which improve post-cooling, likely due to the nonpolar semi-crystalline nature of wax polymers. Similarly, Class IV papers, featuring polyolefin and PVOH-based coatings, also demonstrate initial low seal strengths that significantly increase with extended cooling, indicative of their semi-crystalline nature and the critical role of cooling in their seal performance.

These results offer engineering guidance by demonstrating how polymer composition and thermal behaviour, as detected by DSC, directly influence seal quality. This insight is crucial for designing and optimizing seal processes in packaging applications, where the selection of coated paper based on its DSC profile can enhance packaging integrity. Furthermore, the influences of cool time, seal pressure and, to a lesser extent, seal time significantly impact hot seal strength, aligning with previous research. The critical role of jaw temperature in heat conductive sealing is also affirmed, dictating the efficacy of other seal parameters. Future studies would benefit from examining the quantity of coatings, as well as the composition of paper substrates, in relation to seal performance.

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Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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