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Correlating physical properties of adhesives with process and bonding performance

Martin Rides and Bruce Duncan

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Division of Engineering and Process Control, NPL

ABSTRACT

This report presents a summary of the test methods and results covering density, surface tension and contact angle, rheological properties, tack and bond performance for a range of hot melt adhesives, and a summary assessment of the processibility of those materials.

A number of correlations between physical properties and process and bond performance have been identified. In particular, the following correlations - in some cases strong correlations - have been identified:

- hot melt contact angle with dynamic moduli,
- hot melt contact angle with hot bond strength (tack),
- water drop contact angle with bond strength,
- open time with bond strength,
- adhesive bead width with adhesive failures from the substrate,
- hot bond strength (tack) with no bond separation and cohesive failure,
- melt flow rate data and shear viscosity with molten tack assessment
- rheological properties with spray limit assessment
- surface tension with spray limit assessment

The difficulties encountered in making such correlations are discussed. Sufficient data have been presented here to enable the reader to carry out further correlation analyses, discounting outliers as necessary and focusing on specific property – performance correlations as desired.

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CONTENTS

1 INTRODUCTION

Adhesive bonding is a key joining technology in many industrial sectors. Adhesives are widely used for their ability to join dissimilar materials, with joints that are durable but at the same time unobtrusive and do not detract from the aesthetics of the finished product. However, as the volumes and market values of adhesives used in industry tend to be relatively low, there are few adhesives specialists employed in manufacturing despite the financial risks of problems with the bonding operation. Project MMS9 'Measurements for Efficiency Improvements in Rapid Bonding Systems' of the DTI Measurements for Material Systems programme aims to develop methods for characterising the properties of adhesives and substrates influencing dispensing and bond formation [1].

Systematic approaches, based on understanding the quantitative properties of the materials involved, are needed for designing and setting up optimised bonding processes in order to improve quality or increase throughput. However, these are rarely used as the methods for obtaining such knowledge are lacking. Bonding processes are normally arrived at through a combination of the bonder's experience, material supplier's advice and trial and error. Altering a process normally relies on the experience and skill of the production operator but problems may arise that require a more thorough approach. Adhesives suppliers are often the primary source of advice for the use of adhesives.

In bonding operations it is vital that the adhesive is applied in the correct locations and spreads over roughened surfaces sufficiently to bond. If sufficiently intimate contact is achieved between the two phases, a physical attraction due to inter-molecular forces develops causing the liquid to conform to the surface on a macro and micro scale, displacing air and thus minimising interfacial flaws. Good wettability of a surface and appropriate rheological characteristics are a prerequisite for ensuring good adhesive bonding. Incorrectly applied adhesive can weaken the bond. Hardening of the adhesive, whether due to cooling, drying or cure reactions before bond formation, overspreading or over absorption into permeable materials can weaken the subsequent joint.

The condition of surfaces prior to bonding also has significant implications for the quality of the bonds formed. There are many physical measurement and chemical analysis techniques that can be used for surface characterisation of metals, plastics (including fibre-reinforced plastic composites) and other substrates [2,3]. Surface measurements can focus on physical properties of the surface (e.g. roughness, surface energy or mechanical properties) or the chemical composition of the surface. There is much background information on structural adhesives (e.g. epoxies or acrylics) and metallic surfaces. Therefore, this work concentrates on non-structural materials, such as paperboard and plastic films, and hot melt adhesives - polymeric materials applied to substrates in a molten form that develop mechanical strength on cooling - typically used in packaging that have received comparatively little attention.

Development of measurement methods for characterising the adhesives, substrates and bond performance, carried out as part of the MMS9 project, has been reported elsewhere [3-11]. This report presents a summary of some of those test methods along with a summary of results for a range of hot melt adhesives. Specifically, it covers:

- Density
- Interfacial energy (surface tension by pendant drop and contact angle by sessile drop),
- Dynamic viscosity

- Capillary flow
- Melt flow rate
- Tack
- Bond performance

A subjective assessment of the adhesives' processing performance is also presented. Although the principal objective of the MMS9 project was to development measurement methods for adhesives testing, the data generated through that process has subsequently been used to see whether any correlations exist between the various materials properties, and between materials properties and the adhesives' process and bonding performance. These correlations are reported here.

2 MATERIALS

2.1 HOT MELT ADHESIVES

Hot melt adhesives are thermoplastic polymer based compounds. They are applied as hot fluids, which increase in viscosity and become more rigid as they cool. Hot melt adhesives, first developed for commercial use in the 1950s, have seen significant growth in applications and many advances in technology and application systems. Customers have demanded higher production speeds and wider application across the range of packaging substrates, resulting in greater demands on the performance of these materials.

Two common base polymers are ethylene vinyl acetate (EVA) copolymers and polyethylene (PE) homopolymers: metallocene based hot melts now entering the market. A tackifier resin is added to achieve good hot tack, waxes to reduce viscosity (viscosity must be low at application temperature to allow good substrate wetting, but not too low to allow excessive spreading) and control the setting speed, and stabilisers to prevent charring. A typical formulation for a hot melt packaging adhesive would be [12]:

- Tackifier resin 35-50%
- Polymer 25-35%
- Wax 20-30%.

Packaging hot melts fall into the following broad applications categories:

- General purpose
- Deep freeze
- Heat/creep resistant
- Difficult substrates.

Several hot melt adhesive grades representing the broad application categories used in the packaging industry were supplied by National Starch and Chemical. These have been referred to as "Packaging hot melt adhesives" and are described in Table 1. A selection of "pressure-sensitive" Dispomelt adhesives, also supplied by National Starch and Chemical, have been investigated and these are described in Tables 2. These Tables provide information on the application, typical processing conditions, materials properties, and performance and process suitability of the materials.

2.2 SUBSTRATES

As part of the MMS9 project, a total of 46 substrates were identified for use in the project. A selection of these, listed in Table 3, were used in the bonding assessment reported in Section 4.2.

3 PHYSICAL MEASUREMENTS

In this Section the methods used and the results obtained are described. The tabulated results are either the original data, such as density and interfacial energy, or values determined from original data (e.g. bond performance test data). The latter has been done to enable correlation of the measured properties and process and bonding performance behaviour to be made on a comparable basis.

3.1 DENSITY

The densities of the adhesives were measured using the Archimedes principle in which the apparent weight of an object immersed in a liquid decreases by an amount equal to the weight of the volume of the liquid that it displaces. The decrease in weight of an object of known volume on immersion can be used to calculate the density of the fluid, ρ_l [13]. For a sphere the density of the liquid ρ_l is determined:

$$\rho_l = \frac{m_a - m_i}{\frac{4}{3}\pi r^3} \qquad (1)$$

where m_a is the weight of the ball in air, m_i is the weight of the ball immersed in the adhesive and r is the radius of the ball. The radius of the ball is measured at room temperature and corrected using the known coefficient of thermal expansion for the steel to obtain values for r at each temperature. The accuracy of the technique was checked for distilled water and the measured value was within 0.12% of the reference value.

Using this principle, a steel ball, suspended by a spot-welded Nichrome wire was lowered into a beaker of molten adhesive, Figure 1. The adhesive was allowed to stabilise for approximately 1 hour at temperature to allow any residual air within the melt to escape and temperature gradients to decay. After immersing the ball in the adhesive, the set-up was allowed to stabilise before the weight was recorded. Again, this was to allowing the temperature to equilibrate, but also to allow any viscoelastic effects that might either support the ball or increase its measured weight due to drag to dissipate. The method is presented in greater detail elsewhere [4].

Density measurements were made for the hot melt adhesives at several typical process temperatures of 110 °C, 130 °C and 150 °C, Figure 2 and Table 4. The results are discussed in greater detail in [5]. The data generally exhibit a decreasing density with increasing temperature as expected. Spurious results for Varni-Melt X10 at 110°°C and Novacol 90 at 150°C are being investigated further.

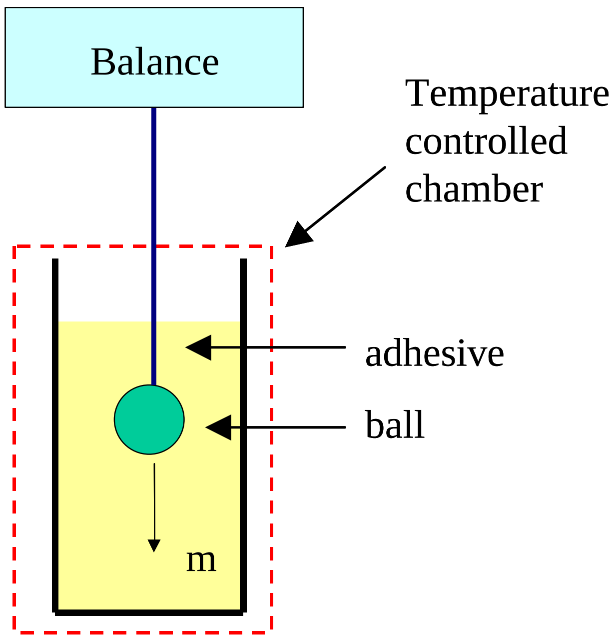

Figure 1: Density measurement by Archimedes' principle

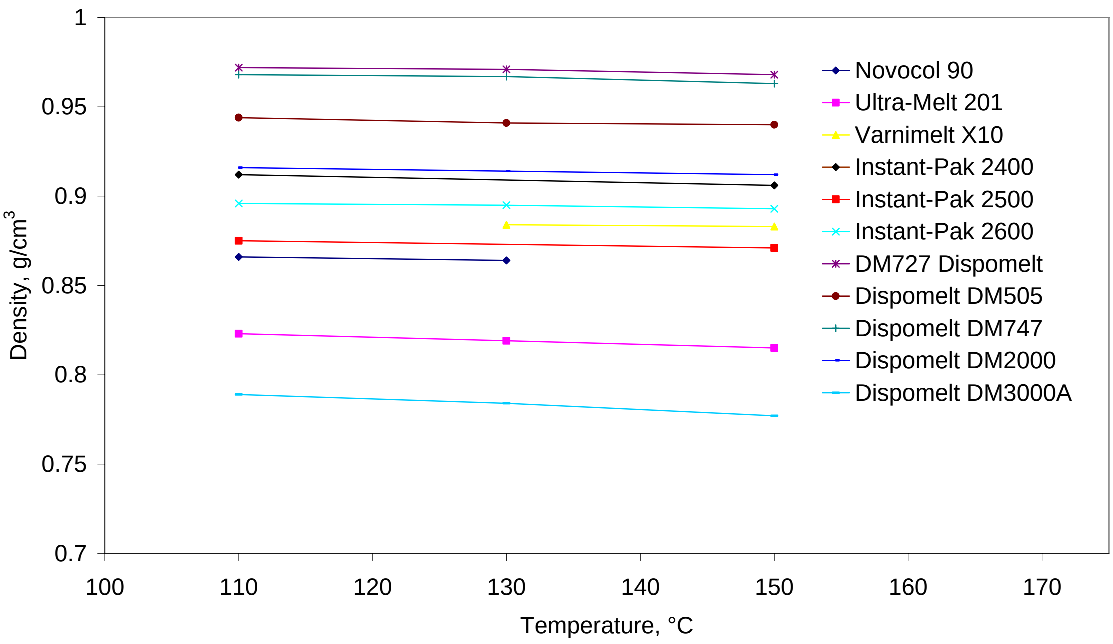

Figure 2: Density values for packaging and Dispomelt hot melt adhesives

3.2 INTERFACIAL ENERGY

The surface tension, or more accurately the liquid-air interfacial tension, of a liquid adhesive is a key property for bond formation [14]:

- Interfacial tension of the adhesive and the surface energy of the substrate determine the wetting of the adhesive on the surface; good wetting promotes intimate contact and higher adhesion.

- The penetration of the liquid into craters and pores in rough and/or porous substrate, which enhances bond strength through mechanical ‘keying’ is a surface tension driven process.

- Surface tension is one of the properties controlling the stability of adhesive jets or sprays during dispensing; it may be an important parameter in effects such as stringing or drop break-up.

The measurement of surface tension properties is therefore considered an important aspect in characterising the properties of hot melt adhesives.

3.2.1 Surface Tension by Pendant Drop Shape Technique

The challenge for determining the properties of hot melt adhesives is that they are only liquid at elevated temperatures and are typically processed at temperatures in excess of 100 °C. The “pendant” drop method provides a means to measure the surface tension of various hot melt adhesives under realistic process temperatures, Figure 3. The equilibrium shape of a hanging pendant liquid drop is due to a balance between the forces acting on the drop. The principal forces are:

- Gravity - which pulls the drop down thereby elongating it; and
- Surface tension – which acts to prevent the growth of surface area and pulls the drop into a spherical shape.

The shape of the drop therefore contains information on both the density and surface tension of the liquid. Building on the work of Laplace, theoretical relationships between shape, density and surface tension have been derived. The Young-Laplace equation [15], which relates the surface tension (γ_l) to the drop shape, is used in automated drop shape analysis (ADSA) techniques:

$$\gamma_l = \frac{\rho_l g X^2}{f\left(\frac{X'}{X}\right)} \qquad (2)$$

where g is the acceleration due to gravity, X is the diameter of the drop at its fullest width and X’ is the diameter of the drop a distance X above the drop base (Figure 3).

The density of the liquid (ρ_l), in this case the adhesive, was unknown at the measurement temperatures and needed to be determined. $f\left(\frac{X'}{X}\right)$ is the shape parameter for the drop and can be calculated by the automated drop shape analysis (ADSA) software or found in look-up tables.

An ADSA technique was used to measure surface tension. The shape of the pendant drop was projected onto the camera using back lighting. The captured video image was analysed using an imaging/software system "CAMTEL, FTA 100 in this work [16], in order to calculate the shape parameter. The samples were enclosed in a temperature controlled chamber to maintain the elevated test temperature.

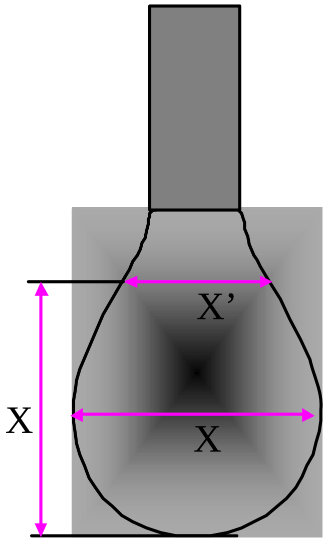

Figure 3: Pendant drop shape

The hot melt adhesive samples were provided in either pellet or block form. It was first necessary to place the adhesive melt into pipettes by cutting the adhesive pellets/blocks into smaller pieces and inserting them into the glass pipettes. A thermocouple was also placed in the pipette to enable accurate measurement of temperature to be made. The adhesive was then melted in the temperature controlled chamber prior to measurement. A plug at the top of the pipette ensured that control of the adhesive drop was maintained during measurements.

Ten readings of surface tension were taken for each packaging adhesive at 110 °C, 130 °C and 150 °C. The Dispomelts adhesives were insufficiently molten at 110 °C, and thus data were obtained at 130 °C and 150 °C only. Data are tabulated in Table 5, and average values presented in Figure 4. It is noted that the Dispomelts exhibited an increasing value in surface tension with increasing temperature, whereas the packaging adhesives exhibited the opposite trend. This may be a real difference, due to the complex chemistry of the materials, or may be due to experimental issues related to the significantly higher viscoelasticity of the Dispomelt adhesives in comparison with the packaging adhesives. The greater viscoelasticity may result in time dependant errors due to the relaxation time behaviour of the material combined with the non-equilibrium nature of the method.

The method and results for the pendant drop measurements are reported and discussed elsewhere in greater detail [4,5].

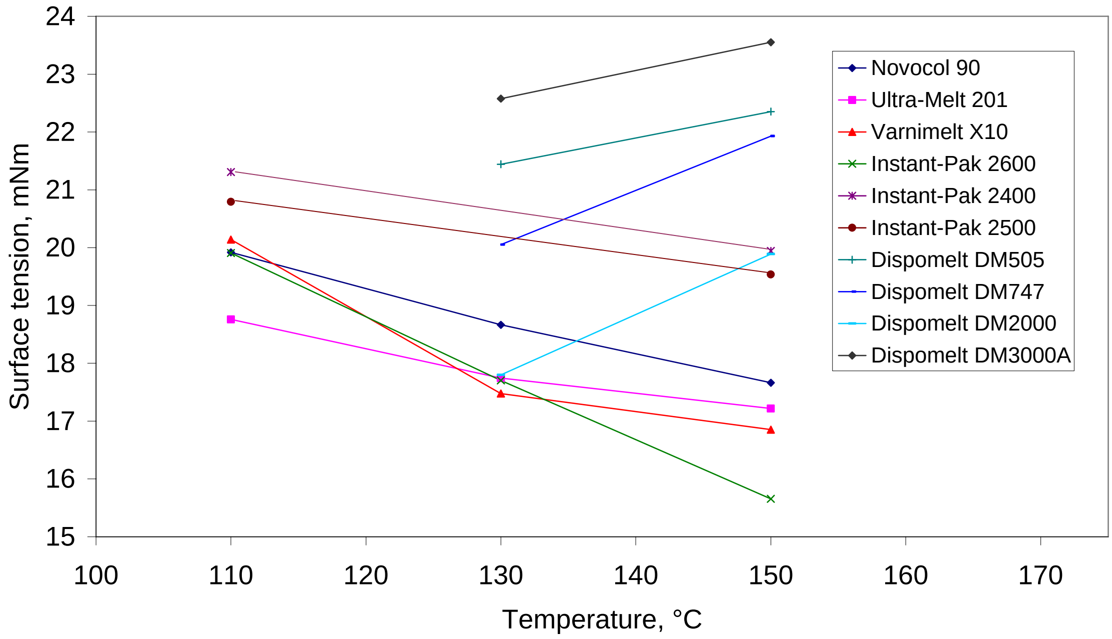

Figure 4: Surface tension results for packaging and Dispomelt adhesives

3.2.2 Contact Angle by Sessile Drop

Contact angles are closely related to wettability [6,7]; the lower the contact angle the greater the wettability. A liquid (adhesive) will wet a solid (adherend) when its surface energy is lower than the solid's surface energy. Force balance or equilibrium at the solid-liquid boundary is given by Young's equation for contact angles greater than zero (see Figure 5):

$$\gamma_{lv} \cos\theta = \gamma_{sv} - \gamma_{sl} \qquad (3)$$

where θ is the contact angle, and $\boldsymbol{\gamma_{lv}}$, $\boldsymbol{\gamma_{sv}}$ and $\boldsymbol{\gamma_{sl}}$ are the surface free energies of the liquid-vapour, solid-vapour and solid-liquid interfaces respectively. The lower the contact angle, the greater the tendency for the liquid to wet the solid, until complete wetting occurs (contact angle $\theta = 0$, $\cos\theta = 1$). For complete wetting to occur, the surface tension of the liquid should be less than or equal to the critical surface tension of the substrate[1]. Conversely, large contact angles are associated with poor wettability.

[1] Critical surface tension is equivalent to the surface free energy, given by $\gamma_{sv} - \gamma_{sl}$

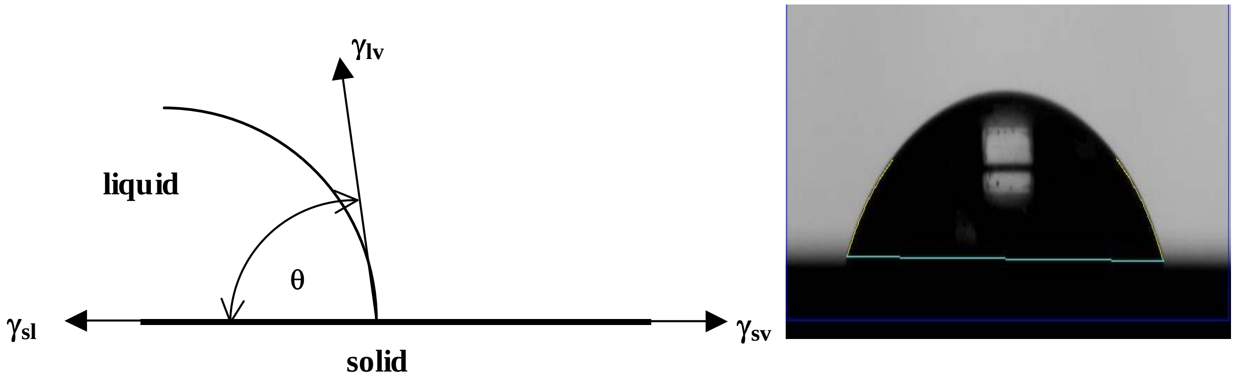

Figure 5: Contact angle of a liquid on a solid surface

The ADSA software and temperature controlled chamber used to measure surface tension were also used to determine contact angles at elevated temperatures. The analysis programme detects the edge of the drop and the surface shape. Numeric algorithms are run to establish the 'shape' of the drop and the slope of the edge in contact with the surface. ADSA is capable, under ideal conditions, of determining contact angles to high accuracy with uncertainties of less than 0.1°.

The contact angle method has been used in two modes. Firstly, using molten hot melt adhesive to form the drop on various substrates, and secondly using water on plate specimens made from the hot melt adhesives.

Measurements of the contact angle of four packaging adhesives (Novacol 90, Varni-Melt X10, Instant-Pak 2400 and Instant-Pak 2500) on one of the surfaces (amorphous polyethylene terephthalate (PET)[2] at 100 °C were made using this apparatus. Despite all these materials having similar, low surface tensions the measured hot melt contact angles vary from 26° (Novacol 90) to 64° (Instant-Pak 2400), Table 6. A difference between the adhesives is that they have different recommended process temperatures: most of these materials are typically dispensed at higher temperatures than the Novacol 90 and consequently have a higher viscosity and are more viscoelastic at 100 °C [8].

The water drop contact angles (WDCA) measurements were performed on flat specimens made from the hot melt adhesives. The results are presented in Table 7.

[2] Surface No 36 in Table 3.

3.3 FLOW PROPERTIES

The rheological properties of the adhesive are key to the manner in which the adhesive is dispensed and subsequently flow before and during bond formation. These adhesives properties can be obtained using various methods.

3.3.1 Dynamic Modulus

Measurements of the shear storage G' and loss G'' moduli of the hot melt adhesives have been made using a Bohlin VORM oscillatory rheometer [8]. Data for the hot melt adhesives at 110 °C and 150 °C are presented in Table 8, obtained from a temperature sweep carried out at 5 °C/minute in cooling using an angular frequency of 5 Hz, a strain of 0.1 and 40 mm diameter parallel plates.

The method and results for the oscillatory rheometry measurements are reported in greater detail elsewhere [8].

3.3.2 Capillary Flow

Capillary extrusion rheometry measurements were performed using a twin-barrel Rosand RH7 extrusion rheometer. Shear viscosity and entrance pressure drop data were obtained at a range of shear rates. For the purposes of correlating with other materials properties and processing performance these data have been reduced to a more manageable dataset. Correlation has been made on the basis of values of shear viscosity and entrance pressure drop values at 9600 s^{-1} and the gradients of the shear viscosity versus apparent shear rate and entrance pressure drop versus apparent shear rate curves. Results are presented in Table 9, along with a description of the shear viscosity and entrance pressure drop parameters in the footnote.

The method and results for the capillary extrusion rheometry measurements are reported elsewhere in greater detail [8].

3.3.3 Melt Flow Rate

Melt flow rate measurements were made using a Ray-Ran Test Equipment Limited 5MPCA Advanced Melt Flow System instrument. Melt volume flow rate (MVR) values were obtained for a range of applied loads from the series 0.325 kg, 1.2 kg, 2.16 kg, 3.8 kg, 5 kg, and 12.5 kg at 150 °C using a standard (ISO 1133) and short length die. The method provides a simple measure of the flowability of the material: the melt volume flow rate (MVR) is defined as the volume of material extruded through the die in 10 minutes.

For the purposes of correlating with other materials properties and processing performance, these data have been reduced to a more manageable dataset. Correlation has been made on the basis of values determined using the 0.325 kg and 3.8 kg loads for both the standard die and short die, and ratios of these parameters. Results are presented in Table 10.

The method and results for the melt flow rate measurements are reported elsewhere in greater detail [8].

3.4 TACK

Tack is the property of an adhesive that determines its ability to instantly form a bond with another surface under light contact pressure. Tack is particularly relevant where bonds must immediately sustain load after assembly. Tack depends on the material properties of the adhesive, the adherend properties and the process conditions. Tack test equipment for characterizing pressure sensitive adhesives was developed in a previous project [17]. This has been modified further to enable measurement of hot melt adhesives, Figure 6. The basic steps of the hot melt tack test are:

- dispense the adhesive onto one of the coupons
- heat the adhesive *in situ* on the coupon to an initial temperature
- bond this coupon to another similar one in under controlled conditions
- separate the bonded coupons in a tensile test under controlled conditions and determine the failure strength of the bond

The tack testing was performed by SATRA. The method and results are reported in detail in [9]. The results have been summarised in Table 11 where hot bond strength as a function of temperature on cooling are presented. Bond strength values at a temperature of 66 °C, referred to as “hot bond strength”, at which data were available by interpolation for all materials, have been determined for purposes of correlation.

Figure 6: Bond strength tack testing

4 PROCESSING PERFORMANCE

4.1 PROCESSING ASSESSMENT

The hot melt adhesives were subjectively assessed by National Starch and Chemical under several categories, namely their suitability and upper speed limit for spray and slot nozzle dispensing for the Dispomelt adhesives, and jet and dauber dispensing for the packaging adhesives. Each category was scored on a 1 to 10 basis, with a score of 10 being awarded for the best performance.

Further information on the processing performance was obtained from the materials datasheets, namely: typical processing temperature, softening point (Dispomelts) or melting point (packaging adhesives), Brookfield viscosity at 140 °C, and for the packaging adhesives the molten tack, open time, and setting time. For the last three parameters a text description of the behaviour was presented in the datasheets. This text description was converted to a numerical value, where 5 corresponds to very high tack, very long open time, and very fast setting time, respectively, for the purposes of the correlation analysis.

This information is presented in Tables 1 and 2.

4.2 BOND MEASUREMENTS

Bond assessment measurements were made by PIRA using their new Pira Adhesive Performance Tester (PAPT II) [3]. A significant quantity of information on the bond performance of numerous adhesive – substrate pairings with variations in both open time and contact time has been obtained using this equipment. An example of the results for Novacol 90 is presented in Table 12. However, quantitative failure load values were not measured by the PAPT II instrument. Thus, to enable correlation with physical properties to be carried out these qualitative data have been reduced to frequency of bond performance in three categories that were considered relevant to the performance of the adhesive, rather than characteristic of the substrates (i.e. fibre tear). These categories were: a) adhesive failure from the substrate, b) cohesive failure of the adhesive, and c) no bond separation. The first is for failure at the interface between the adhesive and the substrate, the second is failure within the adhesive and the last indicates that the PAPT II instrument was not able to break the bond. The frequency of occurrence of these events has been determined using the same subset of adhesive – substrate pairs for each type of failure mechanism. The results of this analysis are presented in Table 13. The frequency of occurrences of each mechanism for each adhesive, expressed as a percentage, was used in the correlation exercise. In such an analysis the effect of test parameters of open time, contact time and substrate type have not been taken into account separately, but summed into a single value for each adhesive. As such they are qualitative measures of the performance of the adhesive.

Furthermore, the compressed bead width of the adhesive was measured for adhesive – substrate pairings. The average value for each adhesive is presented in Table 14 and has also been used in the correlation exercise.

5 CORRELATION OF PHYSICAL PROPERTIES AND PROCESSING BEHAVIOUR

A wealth of data has been collected on the physical properties of the adhesives and also on the process and bonding performance and is presented here and elsewhere in more detail in various reports produced by the project partners of MMS9. These data have been selectively reduced in quantity to enable investigation of correlations between physical properties and process and bonding performance measurements and assessment. It is clear that this data reduction process will remove detail from the data and thus may hide correlations on that scale. Furthermore, in searching for correlations the Dispomelt adhesives and the packaging adhesives have been considered separately, due to the significant differences in their chemistry and consequently in their physical properties. The correlation is based on properties at a specified temperature or other testing conditions, thus materials are compared on a common basis. It is considered, however, that further correlations may exist, particularly on the level of greater detail. Thus considerably more data are presented in this and other reference reports than has been used in the correlation study presented, to enable others to investigate potential correlations. In particular, it is noted that the typical processing temperature differs from material to material. This is expected to have an influence on the correlations. Further correlations could be carried out on the unreduced data sets and also of data at temperatures related to the typical process temperatures.

Initially a correlation analysis was performed using the Microsoft Excel correlation analysis tool as an initial study. Subsequently, correlations were investigated through plotting data and visually assessing whether relationships exist. This approach was taken as it was considered that the Microsoft Excel correlation analysis was sensitive to outliers that might mask correlations and that the visual assessment was more suitable for identifying non-linear relationships between parameters.

A number of correlations have been identified and these are presented below. In the correlations presented below, those relating specific physical properties have been presented first, followed by correlations of physical properties with processing and performance behaviour.

It is expected that there will be correlations between various rheological properties and these are illustrated in Figures 7 – 9[3]. Brookfield shear viscosity results at 140 °C exhibit a good correlation with the shear loss modulus G'' at both 110 °C and 150 °C, as expected. However, the shear storage modulus G', or elastic component of the flow behaviour, shows a poorer correlation. This may, in part, be due to the relatively low elasticity of these materials and consequently difficulties with reliable measurement, particularly at the higher temperature. Similarly, the Brookfield viscosity correlates well with both short and long die melt flow rate data for both 0.325 kg and 3.8 kg loads, and also with shear viscosity and entrance pressure drop data, Figure 8. The melt volume flow rate is a measure of the ease of flow and thus exhibits the opposite trend to shear viscosity.

Similar correlations have been identified between the shear loss modulus G'' determined by oscillatory rheometry with melt flow rate and capillary rheometry values, Figure 9. Again this is not surprising as in both cases the property being considered is the viscous component of flow. However, the test conditions over which data were obtained differ significantly, for

[3] In these and all subsequent correlation plots each point represents a different material.

example the oscillatory measurements were performed in the small strain linear viscoelastic regime, whereas the melt flow rate and capillary extrusion testing are large strain measurements. Furthermore the strain rates in testing may also differ significantly, depending upon the material and test conditions (e.g. 0.325 kg cf. 3.8 kg load in MVR testing).

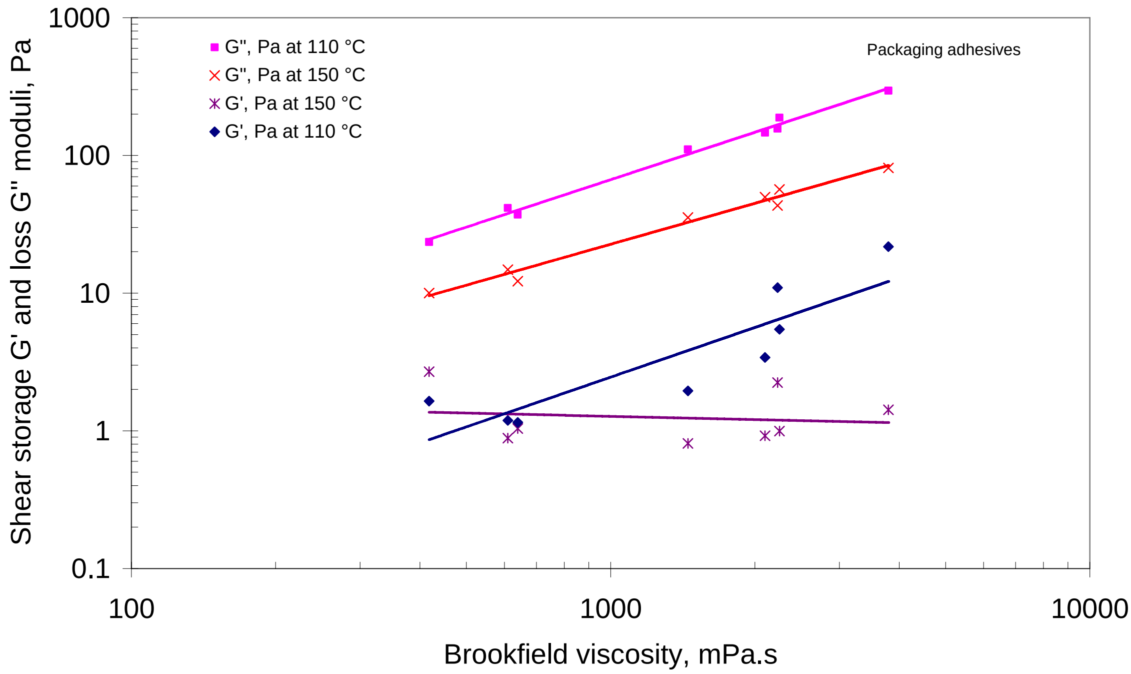

Figure 7: Correlation of rheological properties with the Brookfield viscosity

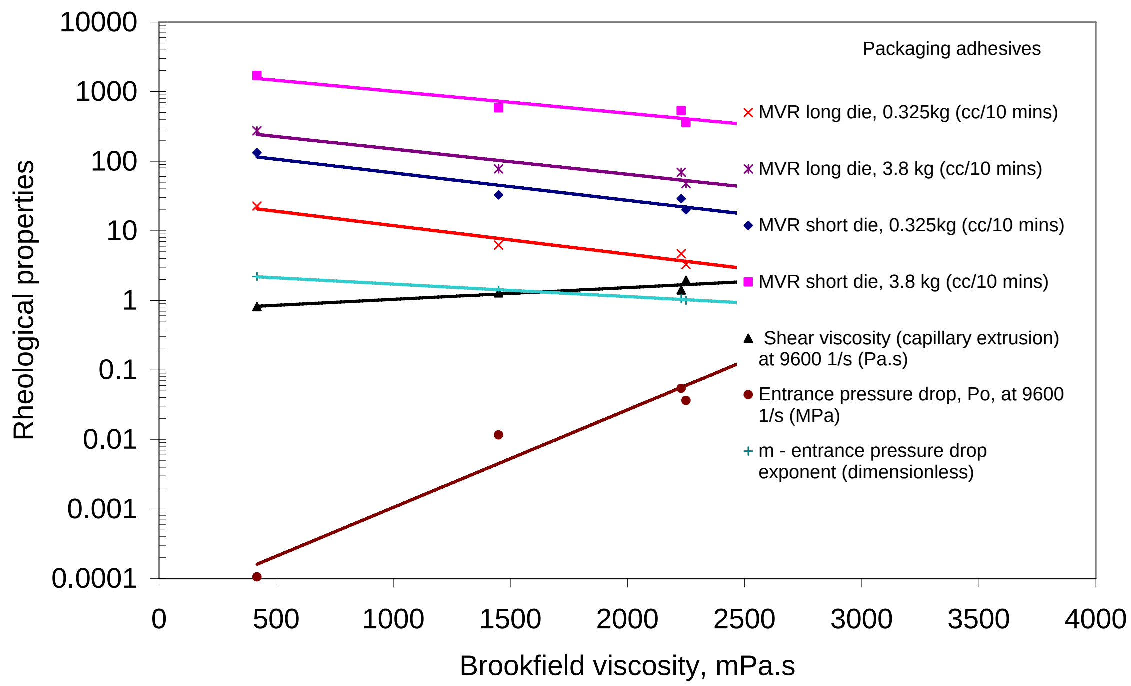

Figure 8: Correlation of melt flow rate and capillary extrusion results with the Brookfield viscosity (for units of rheological properties see legend)

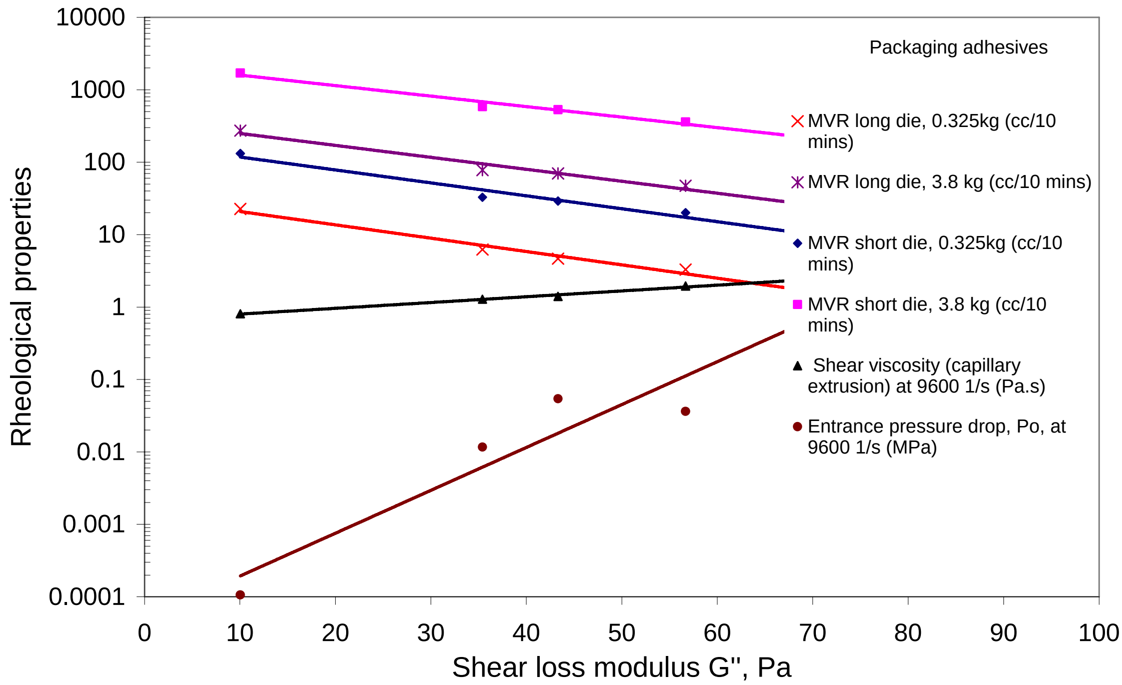

Figure 9: Correlation of melt flow rate and capillary extrusion results with shear loss modulus results (for units of rheological properties see legend)

In terms of the surface energies of the hot melt adhesives, the behaviour was considered to significantly depend upon the measurement method used. A potential correlation was observed between the hot melt contact angle with the water drop contact angle, Figures 10a and 10b. Both these figures indicate a negative correlation between the hot melt contact angle and the water drop contact angle, suggesting that surfaces that are poorly wetted by water are more easily wetted by the hot melt adhesives.

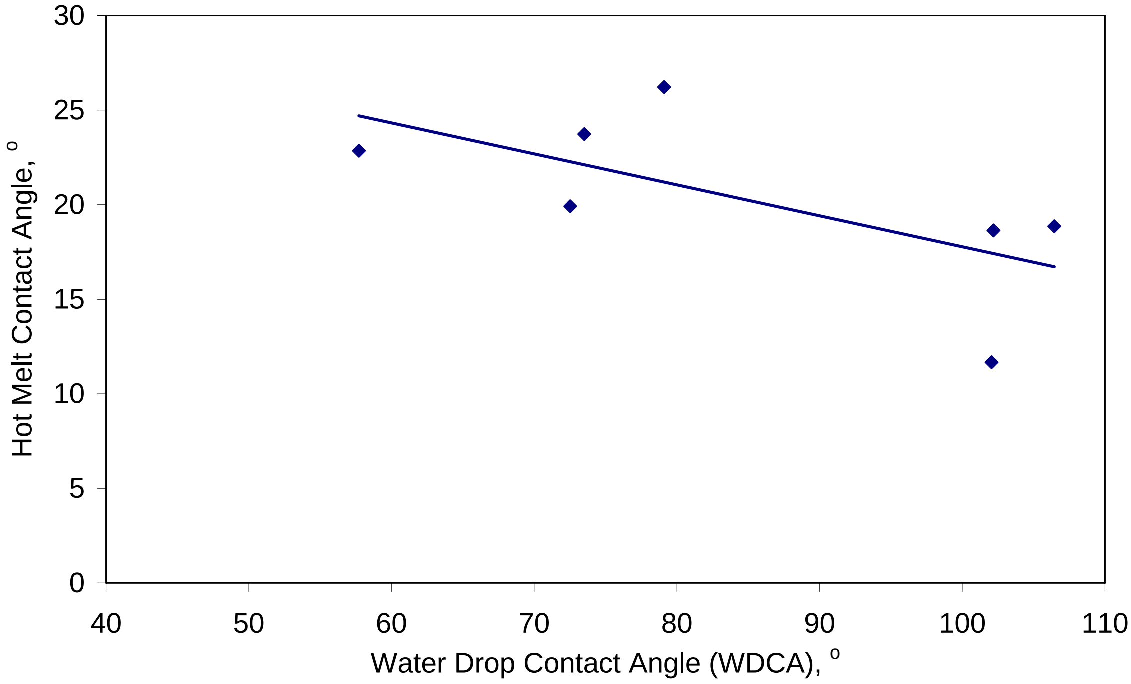

Figure 10a: Relationship between the measured contact angles of a molten hot melt adhesive on a selection of packaging surfaces and the water drop contact angle (room temperature)

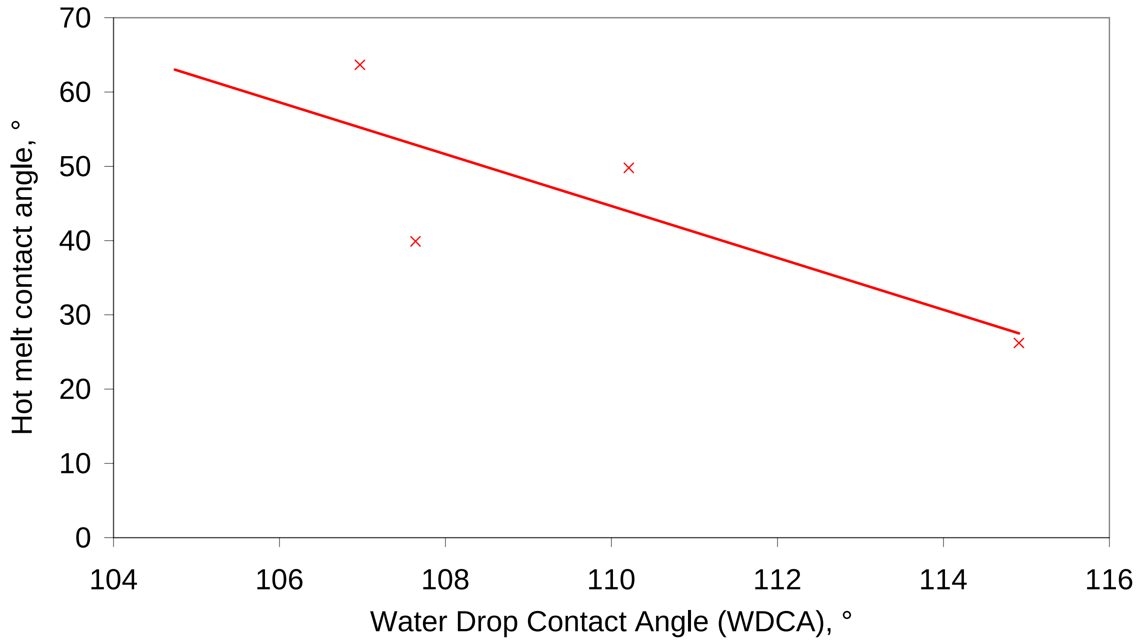

Figure 10b: Relationship between the measured contact angles of several hot melt adhesives on a PET surface at 100 °C with water drop contact angle (room temperature)

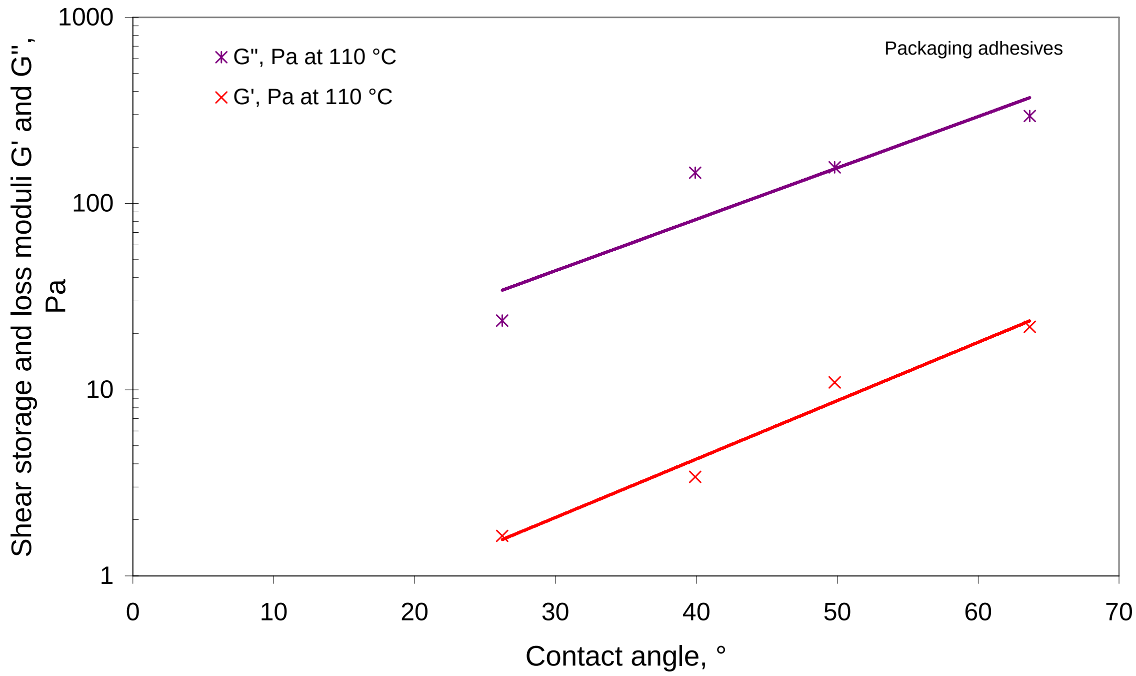

Figure 11: Apparent correlation between wetting (hot melt contact angle) and viscoelastic properties

A correlation between the dynamic moduli (G' and G'') at 110 °C and the measured contact angles for hot melt adhesives at 100 °C on a PET surface at 100 °C is clearly demonstrated in Figure 11. No significant correlation was observed between the surface tension and the hot melt contact angle. This suggests that, at this temperature where the viscosities of adhesives are relatively high, the wetting behaviour is dominated by the viscoelastic properties rather than the surface tension of the adhesives.

The hot melt contact angle and WDCA have both been correlated with hot bond strength (tack), Figure 12, although the variation in WDCA values is small. An increasing bond strength with increasing contact angle is not an expected trend: one would expect a larger bonded area, corresponding to a smaller contact angle, to result in a higher bond strength. However, considering this evidence in conjunction with the moduli – contact angle correlation shown in Figure 11, a larger contact angle corresponds to higher moduli and this larger modulus may be a dominant factor in the contact angle - bond strength correlation shown in Figure 12. For the four materials shown in Figure 11 there is a reasonable correlation of moduli with bond strength, but this was not the case for all the other materials with the Cool Melt and the Instant Pak 2600 materials lying off the correlation curve. It is noted that these materials are lower temperature materials and this is likely to affect the correlation. Hot bond strength has also been correlated with open time, Figure 13. Although there appears to be a relationship between hot bond strength and rheological properties, the explanation for its form is not certain, Figure 14. High melt flow rate behaviour, i.e. lower viscosity, relates to either low or high hot bond strength. The low hot bond strength could be due to low molecular weight components resulting in low strength properties (also resulting in low viscosity), whilst the high hot bond strength could be due to the low viscosity material wetting well the substrate and therefore producing larger bond areas, and consequently strong bonds.

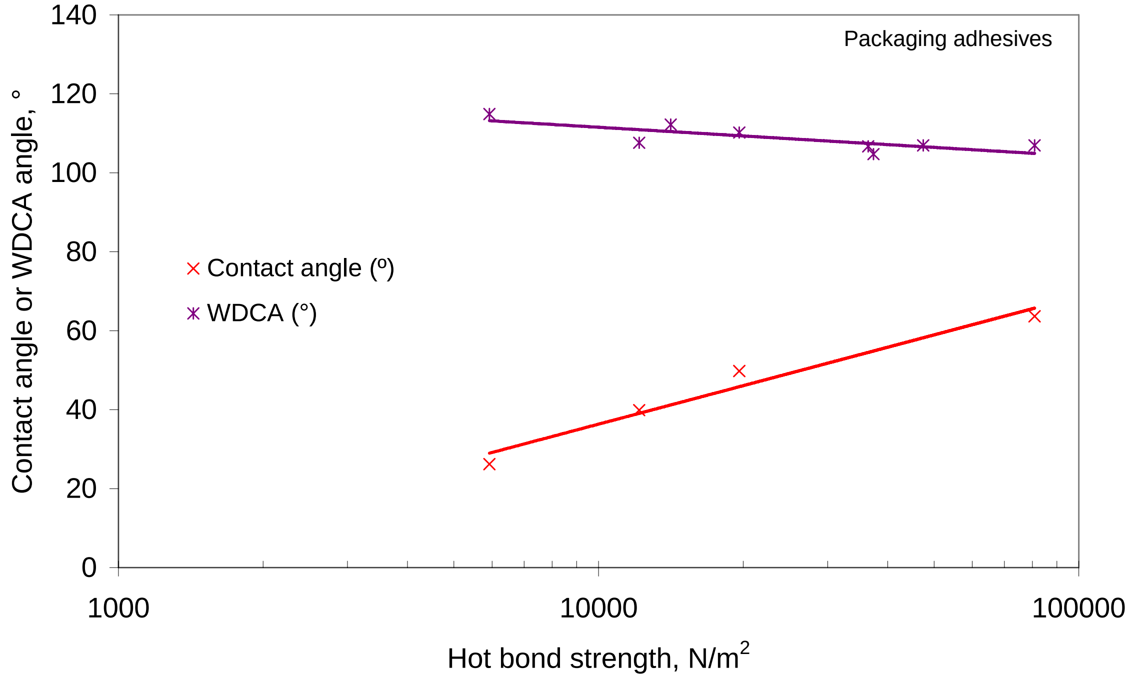

Figure 12: Correlation of hot melt contact angle and WDCA angle with hot bond strength (tack)

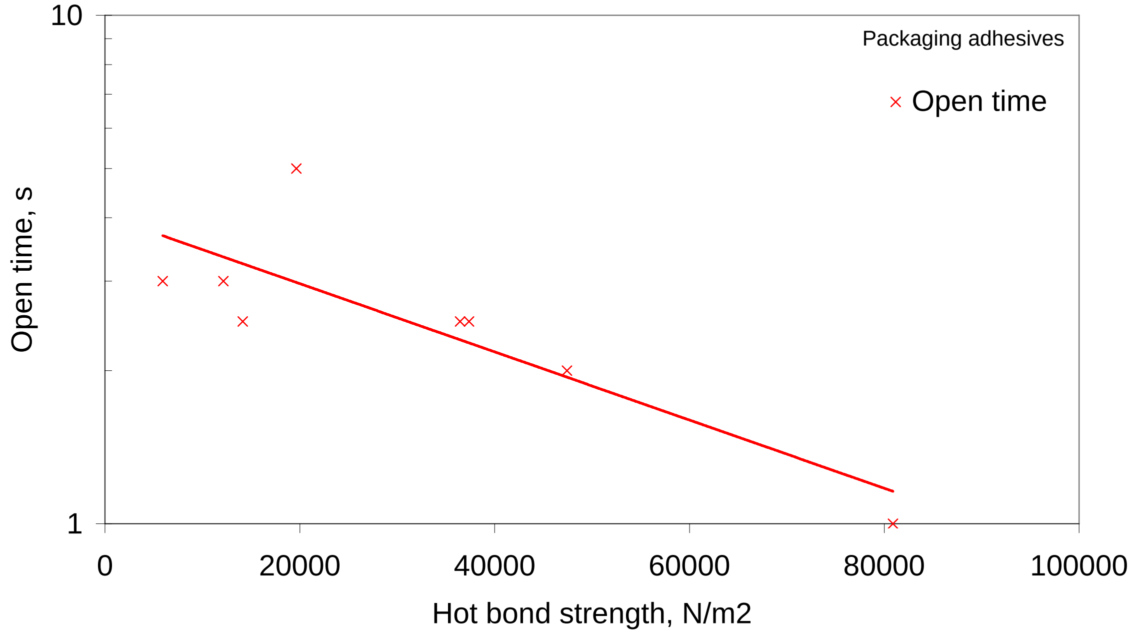

Figure 13: Correlation of open time with hot bond strength (tack)

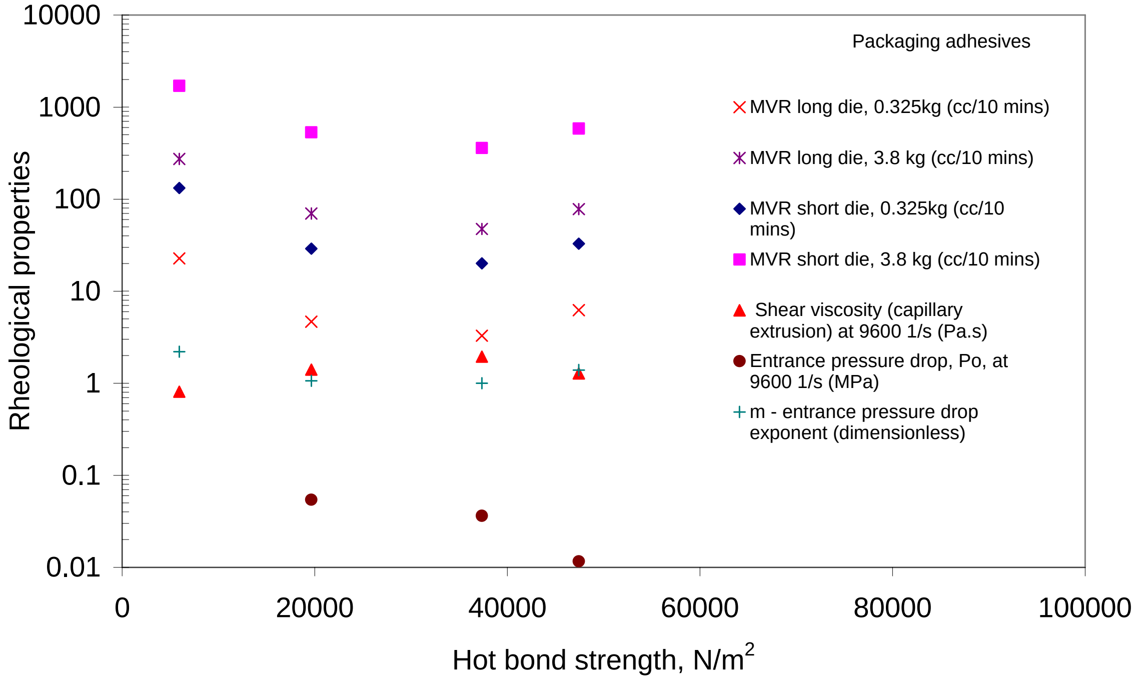

Figure 14: Correlation of rheological properties with hot bond strength (tack)

Of the three categories of bond performance, the adhesive failures from the substrate correlates negatively with the adhesive bead width, indicating that as the bead width increased fewer bonds were failing due to adhesive failure at the substrate, Figure 15. Similarly, there was an increase in cohesive failure and a slight increase in "no bond separation" with increasing bead width. The bond performance measures also exhibit correlations with the hot bond strength (tack) values, Figure 16. Increasing hot bond strength corresponds with increasing frequency of "no bond separation" and decreasing frequency of cohesive failure. However, the effect of hot bond strength on adhesive failure from the substrate is not clear, Figure 16. This latter parameter, adhesive failure from the substrate, is likely to be more dependent on the substrate itself, and as a variety of surfaces were used in the testing it is perhaps not unexpected that a clear correlation was not obtained.

The assessment of molten tack, a parameter specified in the materials' data sheets, correlated well with both the short and long die melt volume flow rate measurements using both the 0.325 kg and 3.8 kg test conditions, Figure 17, and with increasing shear viscosity, Figure 18.

Correlations of physical properties with the process assessment for suitability and upper speed limit for spray and slot nozzle dispensing for the Dispomelt adhesives, and jet and dauber dispensing for the packaging adhesives were not easily apparent. For the Dispomelts it was confused somewhat by the presence of the Dispomelt DM505. On removal of the data for DM505 a correlation of rheological properties with the spray limit assessment for the remaining Dispomelts was apparent, Figure 19. This was not apparent for the slot limit assessment, but this may have been due to the fact that the materials were scored from 7 to 10 for the slot limit assessment (c.f. 0 to 10 for the spray limit assessment) and were thus not sufficiently different for a trend to be observed. Similarly, a correlation was observed for surface tension and the spray limit assessment, Figure 20. A similar trend with suitability for spraying was observed.

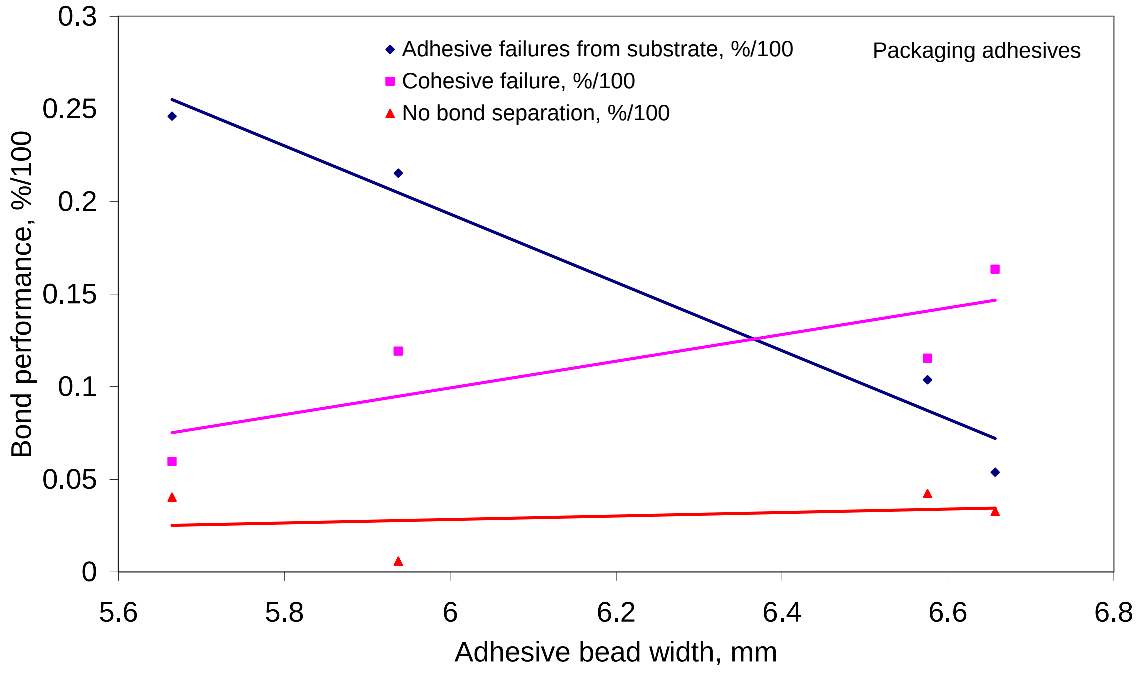

Figure 15: Correlation of bond performance with adhesive bead width

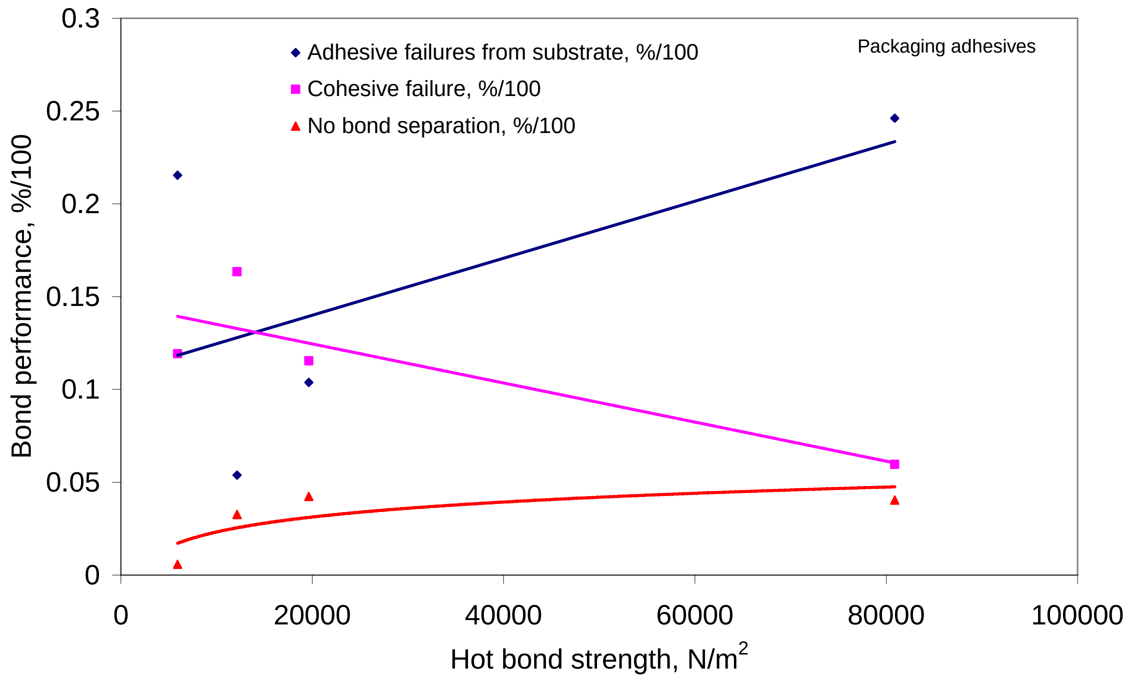

Figure 16: Correlation of bond performance with hot bond strength (tack)

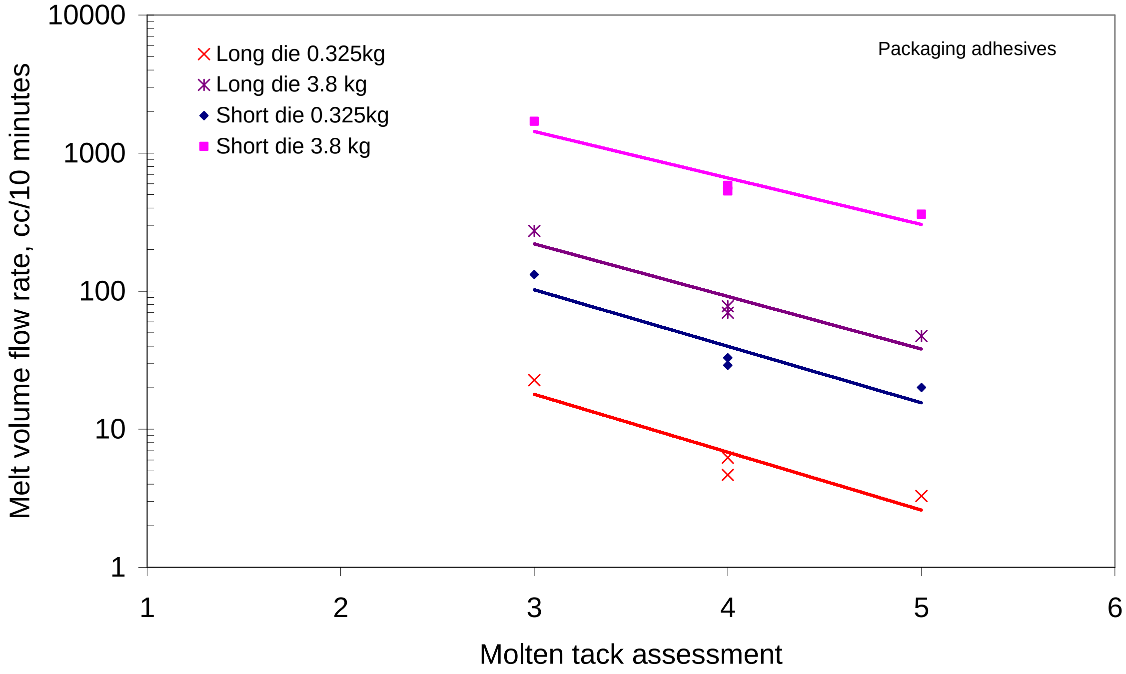

Figure 17: Correlation of molten tack assessment with melt volume flow rate parameters

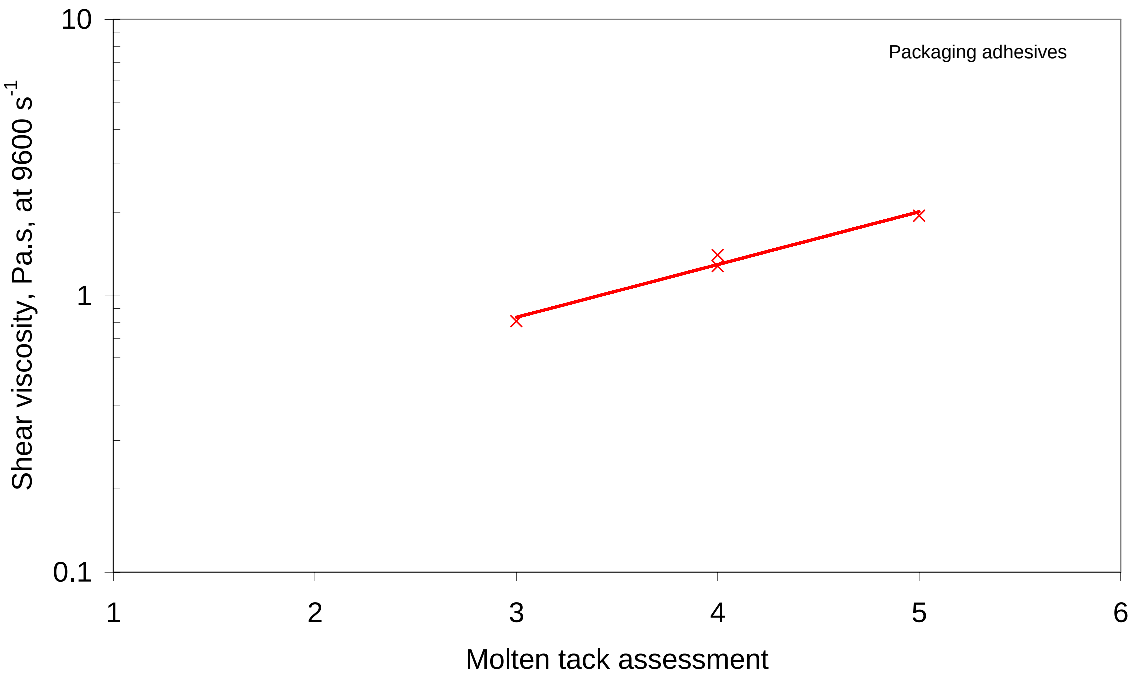

Figure 18: Correlation of molten tack assessment with shear viscosity

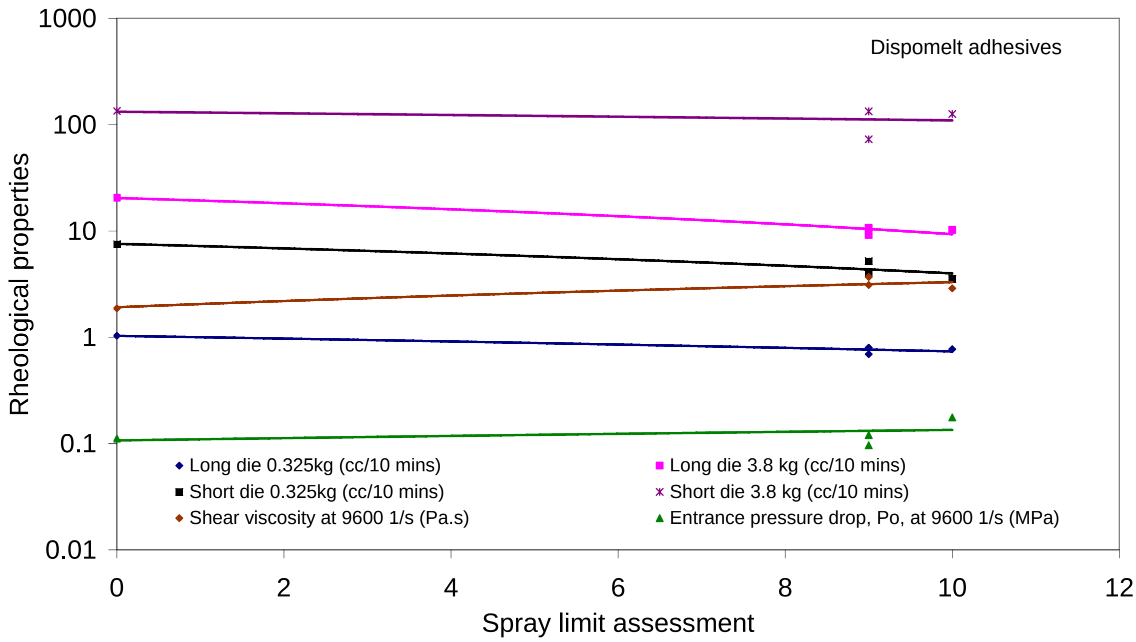

Figure 19: Correlation of spray limit assessment with rheological properties (for units of rheological properties see legend)

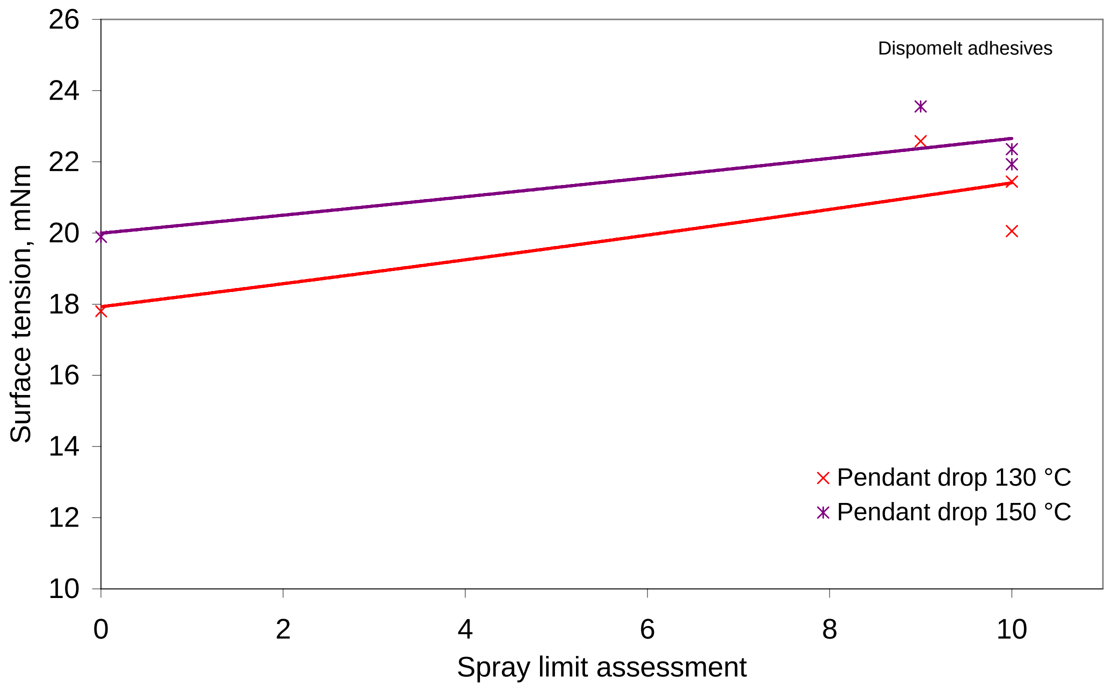

Figure 20: Correlation of surface tension and spray limit evaluation

6 DISCUSSION AND CONCLUSIONS

Data on physical properties, processing performance and bonding performance for a range of packaging and Dispomelt hot melt adhesives, produced largely as a result of technique development and assessment activities in the project MMS9, have been collated and, where necessary, reduced in quantity to enable a correlation analysis to be carried out. The methods used and results for these data are described, and references to other documents for further details are provided.

A number of correlations between the physical properties and process and bond performance have been identified. However, it was considered that correlations were not so likely if data for the Dispomelts and the Packaging adhesives were taken together, due to their significant difference in chemistry and consequently in their properties and process and performance behaviour. Furthermore, it was considered that identification of correlations was made more difficult in the case of the Dispomelt adhesives by the inclusion of the DM505 grade into the analysis. Similar issues might also apply to the packaging adhesives, for example Novacol 90 – a low temperature grade. In assessing these correlations it must be considered that some of the correlations may reflect underlying relationships with other properties, or may just be coincidental. Further correlations might be identifiable if complete data sets for some of the materials were available, in particular for the Dispomelt adhesives. For a more complete correlation exercise, a design of experiments approach is recommended.

Nevertheless a number of correlations have been identified. In particular, the following correlations - in some cases strong correlations - have been identified:

- hot melt contact angle with dynamic moduli,
- hot melt contact angle with hot bond strength (tack),
- water drop contact angle with hot bond strength (tack),
- open time with hot bond strength (tack),
- adhesive bead width with frequency of adhesive failure from the substrate,
- hot bond strength (tack) with no bond separation, and with cohesive failure,
- melt flow rate data and shear viscosity with molten tack assessment
- rheological properties with spray limit assessment
- surface tension with spray limit assessment

These key findings are discussed in more detail in Section 5.

Sufficient data have been presented here to enable the reader to carry out further correlation analyses, discounting outliers as necessary and focusing on specific property – performance correlations as desired.

7 ACKNOWLEDGEMENTS

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Table 1: Processing conditions, datasheet properties and processability assessment: Packaging hot melt adhesives

Hot melt adhesives										Jet application		Dauber application	
NPL code	Adhesive name	Comments & Application method	Typical process temperature, °C	Process temperature, °C	Melting - mid point, °C	Viscosity (Brookfield), mPa.s at given temperature	Molten tack	Open time	Setting time	Suitability for process	Upper limit of processing speed	Suitability for process	Upper limit of processing speed
AQHN003	Novacol 90, 134-823A	Deep freeze (very low application temperature) Jet	100	90 °C in tank, 100 °C at jets	(70)	3080 @ 80 °C 1390 @ 100 °C	Medium	Medium	Medium	10	7	5	5
AQHN006	CM Ultra 100, 134-761A	Standard (low temperature application) General purpose: wheel, jet, dauber	130	120 - 140 °C, typically 130 °C	(95)	1200 @ 120 °C 800 @ 130 °C 610 @ 140 °C 320 @ 160 °C	High	Short-medium	Fast	10	10	10	10
AQHN007	Ultra-Melt 201, 134-265A	Standard General purpose: wheel, jet, dauber	170	160-180 °C, typically 165-170 °C	(95)	2250 @ 140 °C 1250 @ 160 °C 750 @ 180 °C	Very high	Short-medium	Fast	10	8	10	10
AQHN010	Cool-Melt 20, 134-231A	Standard (low temperature application) General purpose	130	120 - 140 °C, typically 130 °C	(95)	1210 @ 120 °C 890 @ 130 °C 640 @ 140 °C	High	Short-medium	Fast				
AQHN012	Instant-Pak 2500, 234-2500	Deep freeze Wheel, jet, dauber	165	160-180 °C, typically 165°C	(95)	2100 @ 140 °C 1200 @ 160 °C 800 @ 180 °C	High	Medium	Medium	10	7	10	10
AQHN013	Instant-Pak 2400, 234-2400	Heat resistant General purpose: not dauber	170	160-180 °C, typically 170-175 °C	(95)	3800 @ 140 °C 1950 @ 160 °C 1100 @ 180 °C	High	Very short	Fast	10	10	1	1
AQHN014	Varni-melt X10	Difficult surfaces Wheel/jet	180	165 °C in tank, 180 °C at jets	(95)	1250 @ 160 °C 750 @ 180 °C	High	Long	Medium	10	8	5	5
AQHN016	Instant-Pak 2600, 234-2600	Standard General purpose: wheel, jet, dauber	165	150-180 °C, typically 160-165 °C	(95)	1450 @ 140 °C 800 @ 160 °C 500 @ 180 °C	High	Short	Fast	10	10	10	10

SUITABILITY FOR PROCESS
Rank 1-10 where 10 is "very suitable for application method", 0 is "will not process by that method"

UPPER LIMIT OF PROCESSING SPEED
Score upper limit of speed of processability, i.e. 10 very high speed, 0 very low (highly unsatisfactory - not economic) speed

Table 2: Processing conditions, datasheet properties and processability assessment: Dispomelt pressure sensitive hot melt adhesives

Dispomelt adhesives							Spray dispensing			Slot nozzle dispensing	
		Application method	Typical process temperature, °C	Process temperature, °C	Softening point – Mettler, °C	Viscosity (Brookfield), mPa.s at given temperature	Suitability for process	Upper limit of processing speed	Comments	Suitability for process	Upper limit of processing speed
AQFW P2	Dispomelt DM727, 134-566A	slot nozzle or spray	160 °C	150-170 °C	90	19000 @ 130 °C 13000 @ 140 °C 8200 @ 150 °C 5400 @ 160 °C 4000 @ 170 °C 2800 @ 180 °C	10	9	enter comments here for spray	8	10
AQLO001	DM505	multiline, coat or spray	softening point 92 °C	140-160 °C	92	1700 @ 160 °C 3800 @ 140 C 12500 @ 120 C	10	10		10	10
AQLO002	DM747	slot nozzle or spray	softening point 89 °C	150-170 °C	89	24700 @ 130 °C 14700 @ 140 °C 9600 @ 150 °C 6700 @ 160 °C 4700 @ 170 °C	8	10	need to apply at high temp.	7	8
AQLO003	DM2000E	nozzle or slot (not spray)	softening point 92 °C	110-140 °C for PE, 130-160 °C for non woven	92	33300 @ 110 °C 12500 @ 130 °C 5900 @ 150 °C 2450 @ 180 °C	0	0	not suitable for spray	9	7
AQLO004	DM3000A		softening point 109 °C		109	16100 @ 130 °C 8050 @ 150 °C 6000 @ 160 °C 3550 @ 180 °C	7	9	need to spray at high temp.	9	8

SUITABILITY FOR PROCESS
Rank 1-10 where 10 is "very suitable for application method", 0 is "will not process by that method"

UPPER LIMIT OF PROCESSING SPEED
Score upper limit of speed of processability, i.e. 10 very high speed, 0 very low (highly unsatisfactory - not economic) speed

Table 3: Substrate description and manufacturer/supplier

Sample No.	Substrate	Manufacturer/Supplier
5	Simcote H/S 440µm/245gsm - fully coated folding boxboard	M-Real Alliance UK Ltd, Simpele Mill, Finland
29	Invercote G 280gsm, 20gsm polyethylene terephthalate coating	Iggesund Paperboard Europe
33	Kraft 190gsm	SCA
34	Test liner II 140gsm	SCA
36	Amorphous polyethylene terephthalate sheet 240µm	Klockner Pentaplast Ltd
45	125gsm printed PET coated bleached Kraft	Unknown/M-real
46	127gsm PE coated waste based paper	Unknown/M-real

Table 4: Density values of hot melt adhesives at elevated temperatures

Adhesive	Density $g.cm^{-3}$		
	110 °C	**130 °C**	**150 °C**
Novacol 90	0.866	0.864	-
Ultra-Melt 201	0.823	0.819	0.815
Varni-melt X10	-	0.884	0.883
Instant-Pak 2400	0.912	-	0.906
Instant-Pak 2500	0.875	-	0.871
Instant-Pak 2600	0.896	0.895	0.893
DM727 Dispomelt	0.972	0.971	0.968
Dispomelt DM505	0.944	0.941	0.940
Dispomelt DM747	0.968	0.967	0.963
Dispomelt DM2000	0.916	0.914	0.912
Dispomelt DM3000A	0.789	0.784	0.777

Table 5: Pendant drop surface tension results

Adhesive	Novacol 90			Ultra-Melt 201			Varni-melt X10		
Test No.	**110 °C**	**130°C**	**150°C**	**110 °C**	**130°C**	**150°C**	**110 °C**	**130°C**	**150°C**
1	19.6	18.18	17.8	18.32	18.05	17.27	19.54	16.94	17.02
2	18.97	18.23	17.77	18.59	17.71	16.67	20.63	17.67	16.59
3	20.19	18.07	17.47	19.51	17.98	17.14	20.02	16.89	17.05
4	19.66	18.66	17.45	18.88	17.98	17.31	20.14	17.37	16.77
5	20.34	19.09	18.13	18.93	17.66	17.36	20.17	17.42	16.79
6	19.77	19.16	17.94	18.87	17.13	17.12	19.39	17.61	16.82
7	19.26	18.94	17.06	18.11	16.98	17.59	21.12	17.39	17.01
8	20.27	18.77	17.12	19.26	18.12	16.92	19.71	17.55	16.83
9	20.11	18.32	18.11	18.63	18.04	17.39	20.77	17.82	16.91
10	21.02	19.22	17.79	18.48	17.79	17.41	19.92	18.11	16.72
Average	19.92	18.66	17.66	18.76	17.74	17.22	20.14	17.48	16.85
St. Dev	0.59	0.44	0.38	0.42	0.40	0.27	0.55	0.37	0.15

Adhesive	Instant-Pak 2600			Instant-Pak 2400		Instant-Pak 2500	
Test No.	**110 °C**	**130°C**	**150°C**	**110 °C**	**150 °C**	**110 °C**	**150 °C**
1	20.19	17.78	15.45	21.82	19.97	20.89	19.06
2	19.26	18.01	15.12	20.29	20.92	19.56	19.79
3	19.78	17.75	15.6	21.53	19.54	19.98	20.38
4	19.71	17.53	16.01	21.22	19.62	20.86	18.74
5	20.61	17.91	15.33	21.81	19.91	20.95	20.73
6	19.54	17.26	15.74	21.55	19.77	21.13	18.97
7	19.93	17.48	15.68	20.78	19.71	21.13	19.04
8	20.11	17.82	16.01	21.13	20.22	21.25	19.52
9	19.96	18.03	15.59	21.56	19.93	20.97	18.97
10	19.97	17.45	16.03	21.38	19.84	21.19	20.15
Average	19.91	17.70	15.66	21.31	19.94	20.79	19.54
St. Dev	0.37	0.26	0.31	0.48	0.39	0.56	0.69

Adhesive	Dispomelt DM505		Dispomelt DM747		Dispomelt DM2000		Dispomelt DM3000A	
Test No.	**130 °C**	**150°C**	**130 °C**	**150°C**	**130 °C**	**150°C**	**130 °C**	**150°C**
1	20.19	22.61	20.19	20.22	18.01	20.09	21.82	21.3
2	21.7	22.63	20.24	21.53	17.63	19.94	22.7	23.35
3	21.12	21.49	20.77	22.22	17.91	20.07	21.85	20.3
4	21.6	21.95	18.97	22.13	17.06	19.37	22.2	24.4
5	21.25	22.77	19.7	22.56	17.45	19.01	21.98	23.73
6	21.89	22.53	19.25	23.56	17.42	20.23	23.01	24.51
7	21.39	22.57	20.05	18.01	17.92	19.76	22.95	23.64
8	22.01	22.71	20.99	22.64	16.59	19.36	22.35	24.38
9	21.7	22.82	18.6	21.56	17.97	21.4	22.77	24.89
10	21.44	22.8	20.89	21.77	17.58	20.96	22.74	23.87
11	21.9	22.94	20.16	23.53	17.47	20.79	22.39	23.44
12	21.16	21.59	20.71	22.87	17.88	17.82	23.01	24.11
13	21.33	22.63	20.01	23.04	18.11	20.57	22.81	23.56
14	21.61	21.76	20.12	21.74	19.97	19.68	22.96	23.22
15	21.35	21.49	20.11	21.55	18.02	19.31	23.12	24.57
Average	21.44	22.35	20.05	21.93	17.80	19.89	22.58	23.55
St. Dev	0.44	0.53	0.69	1.39	0.73	0.88	0.45	1.24

Table 6: Contact angles for different hot melt adhesives on PET at 31 seconds

Adhesive	Contact angle on PET at 100 °C (°)	Typical Process Temperature (°C)	Complex Modulus G* at 110°C (MPa)
Novacol 90	26	100	0.7
Varni-melt X10	50	180	157
Instant-Pak 2400	64	175	296
Instant-Pak 2500	40	165	147

Table 7: Water drop contact angle (WDCA)

Hot melt grade	WDCA (°)	Standard deviation (°)
CM Ultra 100	112.2	1.1
Cool Melt 20	106.7	1.5
Instant Pak 2400	107.0	1.1
Instant Pak 2500	107.6	1.0
Instant Pak 2600	106.9	1.1
Novacol 90	114.9	1.1
Ultra Melt 201	104.7	1.5
Varni-melt X10	110.2	2.7

Table 8: Oscillatory rheometry data

Sample ID	Test ID	G', Pa at 150 °C	G", Pa at 150 °C	tan δ at 150 °C	G', Pa at 110 °C	G", Pa at 110 °C	tan δ at 110 °C
AQFW	boh482	6.78	221.55	32.7	149.91	1440.91	9.6
AQHN 003	boh465	2.69	10.03	3.7	1.64	23.51	14.3
AQHN 006	boh469	0.88	14.84	16.8	1.19	41.52	34.9
AQHN 007	boh470	1.00	56.68	56.9	5.45	188.27	34.5
AQHN 010	boh468	1.04	12.22	11.8	1.15	37.13	32.2
AQHN 012	boh466	0.92	49.73	54.0	3.41	146.45	43.0
AQHN 013	boh467	1.42	81.15	57.2	21.77	295.55	13.6
AQHN 014	boh472	2.24	43.33	19.3	10.94	156.36	14.3
AQHN 016	boh471	0.81	35.40	43.7	1.95	110.55	56.7
AQLO 001	boh483	2.10	94.36	44.9	236.82	1147.27	4.8
AQLO 002	boh484	12.31	243.55	19.8	241.09	1306.36	5.4
AQLO 003	boh485	28.17	184.73	6.6	209.09	661.55	3.2
AQLO 004	boh486	18.11	242.36	13.4	134.91	809.73	6.0

Table 9: Capillary extrusion shear viscosity and entrance pressure drop data

AQFW DM727(ROS225, 0.5 mm)

Shear rate, s^{-1}	P_l, MPa	P_s, MPa	Po, MPa	Shear viscosity, Pa.s
9600	2.028	0.152	0.100	3.137
14400	2.349	0.190	0.129	2.408
19200	2.632	0.241	0.174	1.999
24000	2.891	0.268	0.194	1.755
28800	3.100	0.295	0.216	1.564
33600	3.338	0.353	0.269	1.426
38400	3.595	0.399	0.309	1.336

	Po parameters	Shear viscosity parameters
slope	0.800	-0.619
intercept	-4.202	2.957

Shear rate, s^{-1}	Po - fitted, MPa	Shear viscosity - fitted, Pa.s
9600	0.097	3.105
24000	0.201	1.761
38400	0.293	1.316

AQLO001 DM505 (ROS222, 0.5 mm)

Shear rate, s^{-1}	P_l, MPa	P_s, MPa	Po, MPa	Shear viscosity, Pa.s
19200	1.747	0.103	0.057	1.375
24000	1.976	0.131	0.080	1.234
28800	2.149	0.142	0.086	1.119
33600	2.337	0.161	0.100	1.040
38400	2.649	0.194	0.125	1.027
43200	2.764	0.211	0.140	0.949
48000	2.736	0.220	0.149	0.842

	Po parameters	Shear viscosity parameters
slope	1.045	-0.491
intercept	-5.709	2.241

Shear rate, s^{-1}	Po - fitted, MPa	Shear viscosity - fitted, Pa.s
9600	0.028	1.938
24000	0.074	1.236
38400	0.121	0.982

AQLO002 DM747 (ROS229, 0.5 mm)

Shear rate, s^{-1}	P_l, MPa	P_s, MPa	Po, MPa	Shear viscosity, Pa.s
19200	2.638	0.244	0.177	2.001
24000	2.950	0.262	0.187	1.798
28800	3.160	0.260	0.179	1.616

	Po parameters	Shear viscosity parameters
slope	0.030	-0.525
intercept	-0.875	2.551

Shear rate, s^{-1}	Po - fitted, MPa	Shear viscosity - fitted, Pa.s
9600	0.176	2.888
24000	0.181	1.786
38400	0.184	1.395

AQLO003 DM2000 (ROS226, 0.5 mm)

Shear rate, s^{-1}	P_l, MPa	P_s, MPa	Po, MPa	Shear viscosity, Pa.s
9600	1.264	0.146	0.115	1.869
14400	1.641	0.214	0.174	1.590
19200	2.033	0.304	0.256	1.446
24000	2.404	0.414	0.359	1.331
28800	2.724	0.509	0.447	1.235
33600	3.037	0.592	0.524	1.168
38400	3.281	0.670	0.597	1.092

	Po parameters	Shear viscosity parameters
slope	1.232	-0.380
intercept	-5.860	1.785

Shear rate, s^{-1}	Po - fitted, MPa	Shear viscosity - fitted, Pa.s
9600	0.111	1.870
24000	0.345	1.321
38400	0.615	1.105

AQLO004 DM3000A (ROS224, 0.5 mm)

Shear rate, s^{-1}	P_l, MPa	P_s, MPa	Po, MPa	Shear viscosity, Pa.s		Po parameters	Shear viscosity parameters
9600	2.365	0.187	0.126	3.643	**slope**	0.995	-0.396
14400	3.102	0.254	0.174	3.176	**intercept**	-4.881	2.143
19200	3.724	0.324	0.229	2.843			
24000	4.275	0.401	0.293	2.591	**Shear rate, s^{-1}**	**Po - fitted, MPa**	**Shear viscosity - fitted, Pa.s**
28800	4.801	0.478	0.357	2.410	9600	0.1200	3.697
33600	5.227	0.560	0.4291	2.230	24000	0.2984	2.573
38400	5.690	0.627	0.485	2.116	38400	0.4762	2.136

AQHN003 Novacol 90 (ROS214b, 0.25 mm)

Shear rate, s^{-1}	P_l, MPa	P_s, MPa	Po, MPa	Shear viscosity, Pa.s		Po parameters	Shear viscosity parameters
66723	3.276	0.167	-0.005	0.404	**slope**	2.199	-0.347
100085	4.394	0.248	0.018	0.359	**intercept**	-12.732	1.289
133447	5.376	0.313	0.033	0.329			
166808	6.204	0.379	0.057	0.302	**Shear rate, s^{-1}**	**Po - fitted, MPa**	**Shear viscosity - fitted, Pa.s**
200170	6.943	0.443	0.083	0.281	9600	0.000	0.809
133447	5.244	0.311	0.038	0.320	38400	0.002	0.500
					100000	0.018	0.359

AQHN007 Ultra-Melt 201 (ROS213, 0.25 mm)

Shear rate, s^{-1}	P_l, MPa	P_s, MPa	Po, MPa	Shear viscosity, Pa.s		Po parameters	Shear viscosity parameters
8340	2.041	0.190	0.088	1.921	**slope**	1.000	-0.313
16681	3.484	0.244	0.065	1.682	**intercept**	-5.422	1.535
33362	5.606	0.427	0.140	1.344			
66723	8.611	0.687	0.248	1.028	**shear rate, s^{-1}**	**Po - fitted, MPa**	**Shear viscosity - fitted, Pa.s**
66723	8.631	0.688	0.248	1.031	9600	0.036	1.950
33362	5.726	0.413	0.118	1.379	38400	0.145	1.264
16681	3.559	0.244	0.060	1.721	100000	0.377	0.937
8340	2.082	0.139	0.031	2.018			

AQHN007 0.25 ROS233 Po

Shear rate, s^{-1}	P_l, MPa	P_s, MPa	Po, MPa	Shear viscosity, Pa.s		Po parameters	Shear viscosity parameters
16681	3.982	0.383	0.184	1.868	**slope**	0.799	-0.324
33362	6.326	0.624	0.308	1.480	**intercept**	-4.128	1.630
50042	8.140	0.823	0.418	1.266			
8340	2.400	0.220	0.099	2.264	**Shear rate, s^{-1}**	**Po - fitted, MPa**	**Shear viscosity - fitted, Pa.s**
					9600	0.114	2.193
					38400	0.344	1.400
					100000	0.739	1.027

AQHN014 Varni-melt x10 (ROS211, 0.25 mm)							
Shear rate, s^{-1}	**P_l, MPa**	**P_s, MPa**	**Po, MPa**	**Shear viscosity, Pa.s**		**Po parameters**	**Shear viscosity parameters**
8340	1.497	0.120	0.044	1.430	**slope**	1.064	-0.342
16681	2.522	0.230	0.103	1.190	**intercept**	-5.502	1.510
33362	4.074	0.427	0.225	0.947			
66723	6.342	0.694	0.381	0.733	**Shear rate, s^{-1}**	**Po – fitted, MPa**	**Shear viscosity - fitted, Pa.s**
100085	8.117	1.063	0.672	0.610	9600	0.054	1.406
					38400	0.237	0.875
					100000	0.655	0.631
AQHN016 Instant-Pak 2600 (ROS212, 0.25 mm)							
Shear rate, s^{-1}	**P_l, MPa**	**P_s, MPa**	**Po, MPa**	**Shear viscosity, Pa.s**		**Po parameters**	**Shear viscosity parameters**
8340	1.262	0.077	0.012	1.230	**slope**	1.391	-0.273
16681	2.332	0.153	0.032	1.131	**intercept**	-7.473	1.194
33362	3.911	0.279	0.078	0.943			
66723	6.275	0.492	0.172	0.751	**Shear rate, s^{-1}**	**Po - fitted, MPa**	**Shear viscosity - fitted, Pa.s**
100085	8.092	0.667	0.256	0.642	9600	0.012	1.281
66723	6.274	0.484	0.163	0.752	38400	0.080	0.877
33362	3.936	0.270	0.067	0.952	100000	0.304	0.676
16681	2.341	0.152	0.031	1.136			
8340	1.366	0.077	0.005	1.338			

Shear rate:
All shear rates quoted are apparent shear rates, corrected for entrance effects but not corrected for non-Newtonian velocity profile (Rabinowitsch [18]).

Shear viscosity:
An apparent shear viscosity η_a, corrected for entrance pressure effects, is defined by:

$$\eta_a = \eta_o \left(\dot{\gamma}_a\right)^{n-1} \quad (4)$$

where $\dot{\gamma}_a$ is the apparent shear rate given by:

$$\dot{\gamma}_a = \frac{4Q}{\pi R^3} \quad (5)$$

R is the die radius, Q is the volume flow rate, and η_o and n are constants. In Table 9 for the shear viscosity parameters the slope is equivalent to n-1, and the intercept is equal to log(η_o).

Entrance pressure drop:
The entrance pressure drop P_o is given by a power-law model:

$$P_o = P' \ \left(\dot{\gamma}_a\right)^m$$

where P' and m are constants. In Table 9 for the entrance pressure drop P_o parameters, the slope is equivalent to m, and the intercept is equal to log(P').

Table 10: Melt volume flow rate (MVR) results

Quoted values are MVR, cc/10 min, determined from LDVT output, tested at 150 °C

Long die load, kg	AQHN003	AQHN007	AQHN014	AQHN016	AQFW	AQL0001	AQL0002	AQL0003	AQL0004
0.325	22.65	3.29	4.67	6.22	0.69	2.02	0.77	1.03	0.80
1.2	-	-	-	-	-	6.48	2.53	4.89	2.77
2.16	152.15	24.90	36.48	42.19	-	-	-	-	5.36
3.8	273.36	47.38	69.68	77.98	9.16	23.49	10.23	20.58	10.72
5	392.49	-	-	-	13.31	-	-	-	-
12.5	-	202.30	316.58	-	56.71	153.94	63.67	126.91	45.94
21.6	2014.66	422.75	730.69	703.37	179.99	-	-	-	-

Short die load, kg	AQHN003	AQHN007	AQHN014	AQHN016	AQFW	AQL0001	AQL0002	AQL0003	AQL0004
0.325	132.10	20.06	29.05	32.85	3.98	7.93	3.54	7.52	5.20
1.2	497.27	-	-	-	-	45.77	14.24	34.58	19.56
2.16	-	174.80	-	285.98	-	-	-	-	39.70
3.8	1700.50	360.50	532.71	584.06	133.48	346.01	126.22	134.54	73.05
5	2129.29	-	-	891.14	186.56	-	-	-	107.72
12.5	-	2126.18	2515.21	-	1164.00	1532.09	907.91	867.11	516.81

Long/short ratio, kg	AQHN003	AQHN007	AQHN014	AQHN016	AQFW	AQL0001	AQL0002	AQL0003	AQL0004
0.325	0.171	0.164	0.161	0.189	0.174	0.255	0.218	0.137	-
1.2	-	-	-	-	-	0.142	0.177	-	0.141
2.16	-	0.142	-	0.148	-	-	-	-	0.135
3.8	0.161	0.131	0.131	0.134	0.069	0.068	0.081	0.153	0.147
5	0.184	-	-	-	0.071	-	-	-	-
12.5	-	0.095	0.126	-	0.055	0.100	0.070	0.146	-
Average	0.172	0.133	0.139	0.157	0.092	0.141	0.137	0.146	0.141

Note: If long/short ratio is small then shear dominated behaviour

Table 11: Hot bond strength 'tack' results

DISPOMELT 727	Temperature, °C	86	76	68	54	47	Estimate value at 66°C
Initial temperature, °C	Mean bond strength, N/m^2	39037	65926	107370	321019	372741	136069
80	Std. dev, N/m^2	(1786)	(16124)	(15736)	(68231)	(59415)	-
COOL MELT 20	Temperature, °C	91	85	77	65	57	Est. at 66°C
Initial temperature, °C	Mean bond strength, N/m^2	3778	6815	14111	38481	65519	36451
110	Std. dev, N/m^2	(0)	(321)	(2003)	(321)	(3349)	-
CM ULTRA 100	Temperature, °C	81	71	65	57	49	Est. at 66°C
Initial temperature, °C	Mean bond strength, N/m^2	5741	7037	15556	64630	105000	14136
90	Std. dev, N/m^2	(1219)	(1611)	(2040)	(11119)	(5929)	-
INSTANT-PAK 2400	Temperature, °C	113	102	89	73	62	Est. at 66°C
Initial temperature, °C	Mean bond strength, N/m^2	4333	11926	17222	44074	101926	80889
130	Std. dev, N/m^2	(962)	(501)	(1094)	(7137)	(3592)	-
INSTANT-PAK 2500	Temperature, °C	83	73	66	58	51	Est. at 66°C
Initial temperature, °C	Mean bond strength, N/m^2	3778	6111	12148	29222	56630	12148
90	Std. dev, N/m^2	(0)	(0)	(0)	(0)	(0)	-
INSTANT-PAK 2600	Temperature, °C	96	90	81	69	57	Est. at 66°C
Initial temperature, °C	Mean bond strength, N/m^2	2852	6481	13185	40667	71741	47422
120	Std. dev, N/m^2	(0)	(0)	(0)	(0)	(0)	
ULTRA MELT 201	Temperature, °C	96	85	78	65	57	Est. at 66°C
Initial temperature, °C	Mean bond strength, N/m^2	6667	15407	17741	39000	57000	37365
110	Std. dev, N/m^2	(1347)	(3919)	(3334)	(3508)	(3464)	-
VARNI-MELT X10	Temperature, °C	83	74	67	58	51	Est. at 66°C
Initial temperature, °C	Mean bond strength, N/m^2	7407	11741	18148	43370	77926	19632
90	Std. dev, N/m^2	(2251)	(257)	(2181)	(1283)	(2722)	-
NOVACOL 90	Temperature, °C	66	54	51	45	40	Est. at 66°C
Initial temperature, °C	Mean bond strength, N/m^2	5926	10519	12111	29037	48815	5926
70	Std. dev, N/m^2	(1407)	(849)	(577)	(1339)	(1156)	-

Table 12: Typical bond performance evaluation report

Adhesive : **Novacol 90** **Substrate:** **127gsm PE coated waste based paper**

Applied adhesive to: reverse

Contact time (sec)	0.5		1.0		2.0		5.0	
0.2								
0.5	T	T	T	T				
1.0	T	T	T	T	T	Ta		
1.5	T	T	T	T	T	Ta	1	1
2.0	T	T	T	T	T	T	Ta	T
3.0			T	T	T	T	Ta	T
4.0					T	T	T	1
5.0							T	Ta
Bond width	7 - 6		7 - 6.5		6.5 - 6			

Key

- Fibre tear
- Minor fibre tear
- Surface lift
- Adhesive failure (from the substrate surface)
- Cohesive failure (within the adhesive)

*	PAPT not able to separate bond
T	Top surface failure
R	Reverse surface failure
a	Fibre tear along adhesive centre line only (figure 36)

Table 13: Adhesive bond failure rates

Adhesive failures from substrate		No.	%
Instant Pak 2400	Total occurrence	128	24.6
	Reverse side occurrence	68	26.2
	Top side occurrence	60	23.1
Instant Pak 2500	Total occurrence	28	5.4
	Reverse side occurrence	14	5.4
	Top side occurrence	14	5.4
Varni-melt X10	Total occurrence	54	10.4
	Reverse side occurrence	24	9.2
	Top side occurrence	30	11.5
Novacol 90	Total occurrence	112	21.5
	Reverse side occurrence	61	23.5
	Top side occurrence	51	19.6

Cohesive failure		No.	%
Instant Pak 2400	Total occurrence	31	6.0
	Reverse side occurrence	19	7.3
	Top side occurrence	12	4.6
Instant Pak 2500	Total occurrence	85	16.3
	Reverse side occurrence	53	20.4
	Top side occurrence	32	12.3
Varni-melt X10	Total occurrence	60	11.5
	Reverse side occurrence	36	13.8
	Top side occurrence	24	9.2
Novacol 90	Total occurrence	62	11.9
	Reverse side occurrence	35	13.5
	Top side occurrence	27	10.4

No bond separation		No.	%
Instant Pak 2400	Total occurrence	21	4.0
	Reverse side occurrence	21	8.1
	Top side occurrence	0	0.0
Instant Pak 2500	Total occurrence	17	3.3
	Reverse side occurrence	17	6.5
	Top side occurrence	0	0.0
Varni-melt X10	Total occurrence	22	4.2
	Reverse side occurrence	22	8.5
	Top side occurrence	0	0.0
Novacol 90	Total occurrence	3	0.6
	Reverse side occurrence	3	1.2
	Top side occurrence	0	0.0

Total no. of possible occurrences = 260 per side

Table 14: Bead width measurements

Bead width, mm	Maximum	Minimum	Average
Instant Pak 2400	7.38	2.38	5.66
Instant Pak 2500	7.88	4.13	6.66
Varni-melt X10	8.54	3.70	6.58
Novacol 90	7.25	3.06	5.94