Innovative Hot Melt Adhesive Solutions for “Hard-to-Bond” Substrates
S. Yalvac, L. Chen, T. Gudmundsson, K. Brown, Y. Jin, C. Rickey, A. McLennaghan

Abstract
As consumerism drives demand for quality, functionality, and appeal in packaging; surface treated and coated cardboards have become more fashionable in the packaging industry. Such cardboards are usually coated with bi-axially oriented polypropylene (BOPP) or polyethylene (PE) laminated film or wax on the surface and colored with glossy oil printing. Conventional hot melt adhesives (HMA) have historically demonstrated limited adhesion to these “Hard-to-Bond” (HTB) substrates.

To solve this growing challenge, Dow has developed a maleic anhydride (MAH)-grafted low molecular weight ethylene-octene copolymer, which has shown great potential for achieving bond strengths not possible with other products with similar functionalities. The use of the MAH-grafted copolymer in HMAs dramatically increased the adhesion to hard-to-bond substrates from freezer to microwave temperatures, while still offering many of the well known attributes of Dow’s low molecular weight ethylene-octene copolymers used in adhesives. Renowned attributes include: thermal stability, increased mileage, excellent processability, low maintenance, clarity, and odor free formulations. The MAH-grafted copolymer is also widely applicable, and can be used in formulations utilizing other polyolefin products to impart polarity to the base polymer thus improving adhesion to polar surfaces.

Defining Hard-to-Bond Substrates
Hard-to-bond (HTB) surfaces can be generally categorized as follows:
1. Low surface energy substrates (PE, PP, Teflon, etc).
2. Low porosity or low surface roughness substrates (coated cardboard and cartons)
   a. Paraffin or polyethylene-coated papers for greasy or wet foodstuff (e.g., meat, cheese, etc.)
   b. Papers coated with mold release agents for cooking or baking (e.g., silicone coated)
   c. Papers coated with fluorinated compounds for wet or greasy foodstuff (e.g., confectionery)
   d. Papers card boards or packaging material coated with aqueous acrylic emulsions for wet or greasy foodstuff (e.g., pastries)
   e. Papers or card boards coated with PVDC (e.g., plates)
   f. Functional coatings such as moisture barriers (e.g., BOPP, PET Laminated-soap wraps, detergent boxes)
   g. Improved whiteness or gloss (clay, titanium dioxide, SB emulsions, or starches)
3. Highly recycled paper stock (very low fiber length due to re-pulping)
A recent study by Dow indicated that the HTB substrates are gaining popularity due to a number of reasons [1]. Among them are:

(a) Consumerism and product appeal: Encouraging the purchase of goods and services in ever-greater amounts; eye-catching package design, shelf space attraction.
(b) Functionality: Providing a barrier between the package contents and external elements

The same study also highlighted that the share of the substrate market that is considered as HTB has reached 15-25% of the overall packaging market, sometimes as high a share as 50% (depending on the market sub-segment and geography). A prime example of this is the Chinese market. Chinese customers, for instance, prefer glossy, colorful packaging regardless of the contents. Consequently, the HTB substrate market comprises a larger share.

In line with industry practice, the significantly increased usage of post-consumer ingredients in manufacturing new substrates can contribute to adhesion problems. Such problems arise because the shorter fibers of the recycled substrates do not allow for a good physical anchoring by the adhesive.

Dow recently introduced a maleic anhydride (MAH) grafted ethylene-octene copolymer to specifically offer solutions to counter adhesion problems. Due to its low viscosity and relatively high MAH graft level, it is possible to partially replace some of the base polymer in a HMA formulation to impart chemical bonding in addition to physical anchoring to the substrate. As a result, improved adhesion levels have been achieved on HTB surfaces employing smart formulation methods.

**Experimental**
The new product utilized in this study is AFFINITY™ GA 1000R, a new functional adhesive polymer from The Dow Chemical Company. The hypothesis was to blend a fraction of AFFINITY™ GA 1000R into an AFFINITY™ GA based adhesive to improve adhesion via chemical bonding to the hard-to-bond substrates. Figure 1 illustrates the principle by which MAH-grafted AFFINITY™ GA promotes adhesion.
Materials
The materials used in this study are displayed below in Table 1. The polymers used were AFFINITY™ GA 1900 and AFFINITY™ GA 1000R. The tackifier used was fully hydrogenated hydrocarbon Eastotac H115, supplied by Eastman Chemical Company. The wax used was Sasolwax H1. Sasolwax H1 is a Fischer-Tropsch wax, supplied by Sasol Wax.

Table 1: Polymers used in HMA formulations in this study

<table>
<thead>
<tr>
<th>Material name</th>
<th>Est. MI at 190°C (g/10min)</th>
<th>Viscosity at 177°C(cP)</th>
<th>Density (g/cm³)</th>
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</thead>
<tbody>
<tr>
<td>AFFINITY™ GA 1900</td>
<td>1000</td>
<td>8200</td>
<td>0.870</td>
</tr>
<tr>
<td>AFFINITY™ GA 1000R</td>
<td>660</td>
<td>13000</td>
<td>0.878</td>
</tr>
</tbody>
</table>

HMA Formulations
Components for the HMA compound were weighed into an aluminum can and preheated in an oven for 180°C for one hour. Subsequently, the components in the can were then mixed in a heated block at 180°C for half an hour with a Paravisc style mixer head at 100 rpm.

HMA Testing
Brookfield Viscosity
The melt viscosity of the HMA formulation was measured at 177°C using a Brookfield Viscometer Model DV-III.

Peel and Shear Strength
The peel adhesion failure temperature (PAFT) and shear adhesion failure temperature (SAFT) of the HMA were tested using ASTM D-4498. Four samples were put in a programmable oven then 100g weights for PAFT and 500g for SAFT were attached to the samples. The oven was turned on and heating started from 30°C at a heating rate of 0.5°C/min. The failure time was recorded and the failure temperature can be calculated accordingly.
**Heat Stress**
The heat stress resistance was measured using the “Suggested Test Method for Determining the Heat Stress Resistance of Hot Melt Adhesive”, method T-3006, prepared by the Institute of Packaging Professions. If at least 80% of HMA bonding do not fail, the sample is considered to have passing heat resistance at the test temperature. The maximum passing heat stress resistance temperature was recorded for each HMA sample.

**Fiber Tear**
The percentage of fiber tear of each HMA sample was evaluated on regular cardboard and hard-to-bond substrates at three different temperatures: Room temperature, -17°C and 60°C. The fiber tear results on these two different substrates were recorded.

**Scanning Electron Microscopy (SEM)**
SEM sample preparation: A specimen of the hard-to-bond substrate was cut out with a razor blade. The isolated piece was micromated using a DIATOME Histo knife at -100°C on a Leica UC6 microtome equipped with an FC6 cryo-sectioning chamber. Sections with thickness around 8 micrometers were collected. Both the sections and block face of the film were observed with LEICA DM2500 light microscope.

SEM observation: The samples were coated in Platinum with an Emitech K575X coater. The setting was 20mA and 60 seconds. The Platinum coated samples were put into Nova Nano 630 SEM and observed by ETD detector at an accelerating voltage of 5 KV, with a working distance around 7.0 mm and spot size of 5. The appearance and morphology of both the treated cardboard side and non-treated paper side of the hard-to-bond substrate were observed.

**Fourier Transform Infrared Spectroscopy (FTIR)**
The treated cardboard side of the substrate was directly applied and measured with the ATR method with Perkin Elmer Diamond FTIR. The collection and processing information are:
- Resolution: 4.0 cm\(^{-1}\)
- Filling: 0 level
- Detector: DTGS KBr
- Mirror velocity: 0.2 cm/min
- Scan range: 650 cm\(^{-1}\) to 4000 cm\(^{-1}\)

**Differential Scanning Calorimetry (DSC)**
Samples of the substrate coatings were examined using differential thermal analysis with Dow Method “DSC – Polymer Additives/Characterization @ B-1470 MS Lab Test Method – NYLCOOLHEAT.” For these measurements a DSC Q2000 instrument was employed. Melting peaks were measured using software methods.
Results and Discussions
This work evaluated the properties and performances of partial substitutions of AFFINITY™ GA 1900 with a MAH-grafted low MW ethylene octene copolymer (AFFINITY™ GA 1000R). The target was to use a MAH-grafted copolymer to develop a technical solution for hard-to-bond substrates. A total of five substrates were tested in this study, one from China, and four from South Africa. The substrate from China used in this study was a beverage packaging substrate sent by a local manufacturer as shown in Figure 2. This substrate represents the typical packaging stocks for food and beverage in the Chinese market. According to customers, current HMA solutions based on EVA or AFFINITY™ GA commercial grades are not able to provide good enough bonding to such substrates.

![Figure 2: Beverage Package Substrate from China](image)

The first two substrates from South Africa can be seen in Figures 3 and 4. They are a coated non-printed cardboard and a coated paper used for packaging reams of printer paper, respectively.

![Figure 3: Coated Non-Printed Cardboard from South Africa (Courtesy of Nortec)](image)
The other two substrates from South Africa are both intended for food packaging. The first being a coated paper for bags of wheat flour, and the second a coated paper for bags of maize meal. They can be seen in Figures 5 and 6 respectively.

Figure 5: Wheat Bag from South Africa (Courtesy of Nortec)
The HMA screening criteria in this study were:

- Application viscosity at 177°C
- Heat resistance
- PAFT and SAFT
- Fiber tear on untreated cardboard at room temperature, -17°C and 60°C
- Fiber tear on the China beverage substrate at room temperature, -17°C and 60°C

**Substrate Characterization Results**

SEM was conducted on all five substrates to further understand their topography. In addition, FTIR was conducted on the Chinese substrate and the other substrates were analyzed with DSC to identify the composition of the surface.

**Chinese Beverage Packaging Substrate**

Figure 7 is the SEM topography of the non-printed side of the Chinese substrate. Its surface was very rough with visible porosity at various magnifications. Figure 8 portrays the SEM images of the printed side of the Chinese substrate. The printed side at 300x magnification shows a very flat and smooth surface with few small void-like defects. The topography was still smooth at higher magnifications (shown at 1000x, 2000x and 3000x), while smaller voids became visible as magnification increased.

The laminated film was sectioned for FTIR and SEM investigation. The film was identified as a mono-layer film with thickness about 11 micrometers, shown in Figure 9 and Figure 10. The IR
spectrum of the film structure shown in Figure 11 suggested its main composition to be polypropylene (PP). Due to the non-polar and low surface energy characteristics of the PP lamination on the Kraft board, establishing a good bond is very difficult.

Figure 7: SEM Images of the non Printed Side
Figure 8: SEM Images of the Printed Side

Figure 9: Reflective Bright Field Image of Block Face Sample
South African Printer Paper Packaging substrate

Figure 12 is the SEM topography of the coated surface of the South African printer paper packaging substrate. Its surface is very flat and smooth with only a small amount of defects. The topography was still smooth at higher magnifications up to 2500x and did not indicate any porosity.

Images of the cross-section of the substrate were also taken as seen in Figure 13. Interestingly, a layered structure is visible with a top layer without any porosity covering the relatively porous bottom layer. At higher magnification it can be seen that the top layer is approximately 16 microns thick.
The surface of the substrate was also analyzed via DSC in order to identify the composition of the coating. As seen in Figure 14, a strong peak is observed at 160 °C and a weaker peak at 76 °C. This indicates the presence of polypropylene on the surface and possibly a lower molecular weight waxy material.
Figure 14: DSC of Surface Coating of South African Printer Paper Packaging Substrate

**South African non-printed coated cardboard**

Figure 15 is the SEM topography of the surface of the South African coated but non-printed cardboard substrate. Its surface is relatively rough and clearly porous, especially at higher magnifications.

Images of the cross-section of the substrate were also taken (as seen in Figure 16). There is a slight indication from these images that there is a bulk of cardboard with a very thin coating on the surface. At the highest magnification there is an indication of a continuous coating, this however, does not agree fully with the topographical images.

Figure 15: SEM images of South African Coated Non-printed Cardboard
The surface of the substrate was also analyzed via DSC in order to identify the composition of the coating. As showcased in Figure 17, only one weak peak is observed at 79 °C, indicating a lower molecular weight waxy coating, likely a paraffinic wax.

**Figure 16: SEM Images of Cross-section of South African Coated Non-printed Cardboard**

**Figure 17: DSC Curve of Coating of South African Coated Non-printed Cardboard Substrate**

**South African Flour Packaging substrate**

Figure 18 is the SEM topography of the surface of the South African Flour Bag substrate. Images were taken across the interface between the printed and non-printed portions of the substrate.
The surface is relatively smooth, although some porosity is clearly evident. No significant difference is observed between the printed and non-printed surfaces on this substrate.

Images of the cross-section of the substrate were also taken as seen in Figure 18. There is no indication of a layered structure, only a dense bulk with a slightly roughened surface.

Figure 18: SEM Images of South African Flour Bag Substrate

Figure 19: SEM Images of Cross-section of South African Flour Bag Substrate

The surface of the substrate was also analyzed via DSC in order to identify the composition of the coating. As exhibited in Figure 20, only one weak peak is observed at 74 °C, indicating a lower molecular weight waxy coating, possibly a paraffinic wax.
**Figure 20:** DSC Curve of Coating of South African Flour Bag substrate

**South African Maize Meal packaging substrate**

Figure 21 is the SEM topography of the surface of the South African Maize Bag substrate. Images were taken across the interface between the printed and non-printed portions of the substrate. The surface is very smooth and continuous on the printed surface, while the non-printed areas are clearly much rougher and more porous.

Depicted in Figure 22, are images of the cross-section of the substrate. Upon close inspection, one can deduce that there is no indication of a layered structure, only a dense bulk with a relatively rough top surface.

**Figure 21:** SEM Images of South African Maize Bag Substrate
The surface of the substrate was also analyzed via DSC in order to identify the composition of the coating. As denoted in Figure 23, only one weak peak is observed at 73 °C indicating a lower molecular weight waxy coating, possibly a paraffinic wax.

**MAH-grafted substitutions: formulations and performances**

Table 2 shows the properties of the HMA formulations used in this study, including test results performed with the Chinese substrate. These model formulations, with 40 wt% of polymer were tested as the functional polymer content was gradually increased from 0 to 40 wt% in 10% increments, until MAH grafting polymer was as the sole polymer (40 wt %) in formulation.
These formulations were applied on regular cardboard and the Chinese hard-to-bond substrate at 177 °C. The viscosities of all these formulations were between 500 and 1500 cps, a requirement for optimal dispensing of the HMA. The incumbent EVA based HMA was shown to have very limited adhesion on the difficult to bond substrate at room temperature and freezer temperature. It could only achieve 34 % and almost 0 % fiber tears at -17 °C and room temperature, respectively. AFFINITY™ GA 1900 alone without MAH grafting had marginal bonding on regular cardboard at -17 °C and 60 °C. Furthermore, it also failed on hard-to-bond substrates, with fiber tear around 60% at both temperatures. By substituting 10 wt% of AFFINITY™ GA 1900 with AFFINITY™ GA 1000R, the adhesion on the hard-to-bond substrate was significantly increased. In addition to increased adhesion, greater resilience to heat emerged as a further benefit. The highest heat resistance was achieved with 30 wt% and 40 wt % of AFFINITY™ GA 1000R in the formulation, passing the 60°C heat stress test. Though the PAFT and SAFT results did not follow the same trend observed in heat stress tests, heat stress tests are considered to reflect a more realistic rendering of real life heat loads on the flaps.

Overall, compared with incumbent EVA and AFFINITY™ GA based adhesives, the MAH-modified adhesive showed a significant improvement on adhesive properties, such as heat stress, fiber tear on regular cardboard and the hard-to-bond substrate. With 20 wt%, 30 wt% and 40 wt% of AFFINITY™ GA 1000R, the formulations reached a sufficient level to ensure commercial success on such substrates.

Table 2: Properties of the HMA Used in the Study with and without MAH-grafted Polymer

<table>
<thead>
<tr>
<th>40% Polymer</th>
<th>34.5% Eastotac H115</th>
<th>25% Sasolwax H1</th>
<th>0.5% ppm I1010</th>
<th>Viscosity @177°C, (cP)</th>
<th>SAFT (°C)</th>
<th>PAFT (°C)</th>
<th>Fiber Tear (%) China Substrate</th>
<th>Fiber Tear (%) Regular Cardboard</th>
<th>Heat Stress Regular Cardboard (failure = cohesive)</th>
</tr>
</thead>
<tbody>
<tr>
<td>EVA</td>
<td></td>
<td></td>
<td></td>
<td>40%</td>
<td>592.87</td>
<td>91.7</td>
<td>57.1</td>
<td>62, 66, 96</td>
<td>-17°C: 75, 100, 67: 5, 3</td>
</tr>
<tr>
<td>AFFINITY™ GA1900</td>
<td></td>
<td></td>
<td></td>
<td>5 wt% AFFINITY™ GA 1000R</td>
<td>589.87</td>
<td>92.4</td>
<td>57.9</td>
<td>71, 59, 82</td>
<td>-17°C: 100, 60°C: 98, 6: 1</td>
</tr>
<tr>
<td>AFFINITY™ GA 1000R</td>
<td></td>
<td></td>
<td></td>
<td>10 wt% AFFINITY™ GA 1000R</td>
<td>590.87</td>
<td>91.7</td>
<td>57.8</td>
<td>75, 100, 96</td>
<td>-17°C: 100, 60°C: 100, 100: 5, 0</td>
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<tr>
<td>AFFINITY™ GA 1000R 1</td>
<td></td>
<td></td>
<td></td>
<td>20 wt% AFFINITY™ GA 1000R 1</td>
<td>601.87</td>
<td>91.3</td>
<td>57.3</td>
<td>100, 100, 99</td>
<td>-17°C: 100, 60°C: 100, 100: 6, 3</td>
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<tr>
<td>AFFINITY™ GA 1000R 1</td>
<td></td>
<td></td>
<td></td>
<td>30 wt% AFFINITY™ GA 1000R 1</td>
<td>601.87</td>
<td>90.7</td>
<td>57.7</td>
<td>100, 90, 87</td>
<td>-17°C: 100, 60°C: 90, 80: 4, 2</td>
</tr>
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</table>
The below table displays the test results using the optimum formulation as based on results with the Chinese substrate. The AFFINITY™ GA 1900 based formulation performed equally well as the MAH-grafted formulation at room temperature and elevated temperatures. At freezer temperature, the MAH-grafted copolymer formulation clearly performed much better.

**Table 3: HMA properties on South African Substrates with and without MAH-grafted copolymer**

<table>
<thead>
<tr>
<th>Formula</th>
<th>Percent Fiber Tear</th>
</tr>
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<tbody>
<tr>
<td>40% Polymer + 34.5% 115°C Tackifier + 25% Fischer-Tropsch Wax</td>
<td>-25°C</td>
</tr>
<tr>
<td>0.670 g/cc, 8,300 cps ethylene-octene copolymer</td>
<td>Blank coated cardboard (Brazil)</td>
</tr>
<tr>
<td>20% MAH-g 13,000 cps ethylene-octene copolymer</td>
<td>Blank coated cardboard (Brazil)</td>
</tr>
<tr>
<td>0.670 g/cc, 8,300 cps ethylene-octene copolymer</td>
<td>SAPPi Printer Paper packaging</td>
</tr>
<tr>
<td>20% MAH-g 13,000 cps ethylene-octene copolymer</td>
<td>SAPPi Printer Paper packaging</td>
</tr>
<tr>
<td>0.570 g/cc, 8,200 cps ethylene-octene copolymer</td>
<td>Flour Bag (South Africa)</td>
</tr>
<tr>
<td>20% MAH-g 13,000 cps ethylene-octene copolymer</td>
<td>Flour Bag (South Africa)</td>
</tr>
<tr>
<td>0.670 g/cc, 8,200 cps ethylene-octene copolymer</td>
<td>Maize Meal Bag (South Africa)</td>
</tr>
<tr>
<td>20% MAH-g 13,000 cps ethylene-octene copolymer</td>
<td>Maize Meal Bag (South Africa)</td>
</tr>
</tbody>
</table>

**Comparative study on formulations with AFFINITY™ GA 1000R and MAH grafted wax**

To further understand the performance differences between different MAH grafted solutions, two formulations were designed as seen below. The first formulation was composed of 40 wt% of AFFINITY™ GA 1900 and 10 wt% of A-C575P, a MAH-grafted wax from Honeywell Corporation. The second formulation was composed of 20 wt% of AFFINITY™ GA 1900 and 20 wt% of AFFINITY™ GA 1000R. Both formulations contained a similar content of 0.22 wt% of MAH groups, either from functionalized polymer (AFFINITY™ GA 1000R) or a functionalized wax (A-C575P). As shown in Table 4, with the same MAH content in the formulation, the MAH grafted polymer brings an overall better performance than the grafted wax, especially for high temperature adhesion, heat stress and flexibility properties. Compared
with A-C575P, AFFINITY™ GA 1000R, it has demonstrated a 7.5 °C increase in heat stress performance. It also outperforms in flex mandrel tests at both room temperature and freezer temperature. AFFINITY™ GA 1000R is also a cost conservative solution.

Table 4: Performances of MAH grafted polymer vs. MAH grafted wax

<table>
<thead>
<tr>
<th></th>
<th>SAFT</th>
<th>PAFT</th>
<th>Fiber Tear US Cardboard</th>
<th>Heat Stress</th>
<th>Flex Mandrel (3mm)</th>
<th>Flex Mandrel (6mm)</th>
<th>Flex Mandrel (15mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Viscosity @177°C</td>
<td>°C</td>
<td>-17°C</td>
<td>RT</td>
<td>60°C</td>
<td>50°C</td>
<td>55°C</td>
</tr>
<tr>
<td>40wt% GA1900, 10wt% A-C575P, 15wt% Sasolwax H1, 34.5wt% Eastotac H100, 0.5wt% I1010</td>
<td>909</td>
<td>69</td>
<td>42</td>
<td>100</td>
<td>100</td>
<td>70</td>
<td>20</td>
</tr>
<tr>
<td>20wt% GA1900, 20wt% AFFINITY™ GA 1000R, 25wt% Sasolwax H1, 34.5wt% Eastotac H100, 0.5wt% I1010</td>
<td>789</td>
<td>83</td>
<td>50</td>
<td>100</td>
<td>100</td>
<td>92</td>
<td>6</td>
</tr>
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</table>

Finally, Figure 24 shows the initial solid and molten colors of the standard and AFFINITY™ GA 1000R modified adhesives. It can be discerned that MAH-grafted polymer modification has very little impact on the adhesive color.

Figure 24: Solid and molten colors of the original and AFFINITY™ GA 1000R modified HMAs

Conclusions
Despite the success of AFFINITY™ GA polymers in packaging adhesives, there have been occasional pockets of applications where the adhesion to certain substrates has been challenging. This paper has presented solutions for adhesion to such hard-to-bond substrates by incorporating MAH-grafted low molecular weight ethylene-octene copolymer (AFFINITY™ GA 1000R) into HMAs based on AFFINITY™ GA1900. Overall, the MAH-modified adhesive showed a significant improvement in inherent adhesive properties, such as: heat stress, fiber tear on regular
cardboard and typical hard-to-bond substrates from diverse global regions and markets. With 20 wt% 30 wt%, 40 wt% of AFFINITY™ GA 1000R, the formulations were good enough to guarantee commercial success on such substrates. Compared with incumbent MAH-g wax, MAH-g low MW ethylene octene copolymer (AFFINITY™ GA 1000R) brings overall better performance, especially when considering high temperature adhesion, heat stress and flexibility. As an added bonus, AFFINITY™ GA 1000R is also a cost-friendly solution.

References

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