A model for hot tack behavior in ethylene acid copolymer films

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ABSTRACT: A model has been developed for hot tack behavior in ethylene methacrylic acid and ethylene acrylic acid copolymers based on statistical regression of data. This model shows trends and provides insights on the factors that influence hot tack strength. A correlation of eight independent variables with hot tack strength showed that the two factors with the greatest impact on hot tack strength are seal temperature and acid content of the film. The melt indices, melt point temperatures, and synthesis temperatures of the film resin had insignificant correlations with hot tack. No significant difference in hot tack strength was found between acrylic and methacrylic acid copolymers. This model provides a better understanding of an important phenomenon in packaging applications, and it can be used to approximate hot tack behavior in acid copolymers when certain variables are specified.

KEYWORDS: Acrylic acid, copolymers, ethylene, film, methacrylic acid, models, tack.

Hot tack is the capability of a heat seal to hold together, when pulled apart, immediately after the heat sealing operation. Hot tack strength is the measure of the maximum stress that can be applied before the seal peels apart. This is different than seal strength, which is a measure of the strength of the seal after it has cooled. Typically, hot tack strength is lower than seal strength.

Hot tack is important in packaging applications. A high hot tack strength at lower temperatures often allows packaging manufacturers to increase line speeds and thereby reduce costs. Hot tack is also a constraining factor in determining the weight of material that can be packaged in a form-fill-seal machine. High hot tack is desirable in cases where bulky products tend to resist package edge sealing, where vibration or cutting takes place while the seal is hot, or where packages are filled hot.

Ionomer resins are regarded by the packaging industry as the materials of choice for best hot tack performance. Acid copolymers are generally recognized as the next best alternative. There are many applications where the less costly but inferior hot tack strength of acid copolymers provides adequate performance. One of the key attributes of acid copolymer resins is their high hot tack performance relative to cost.

However, hot tack is a poorly understood phenomenon. The purpose of this study was to gain a better fundamental understanding of hot tack, and perhaps to find key factors that can be controlled to influence hot tack strength. Refer to the "Literature cited" section for more background information and previous studies on hot tack.

Discussion

Experimental strategy

In this study, hot tack strength was measured for 17 ethylene methacrylic acid (MAA) and ethylene acrylic acid (AA) copolymers, with acid levels ranging from 4 weight percent (wt.%) to 15 wt.%, and melt indices ranging from 1.5 decagrams per minute (dg/min) to 25 dg/min. These resins are listed in **Table I**.

The acid contents of the acrylic acid resins were normalized for equivalent mole weight percent of methacrylic acid because methacrylic acid has a greater molecular weight than acrylic acid. This was done so that the effects of methacrylic acid could be compared with the effects of acrylic acid on a molar basis.

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Variables tested

The following variables and their combinations were tested for correlation with hot tack strength:

- Acid content
- Seal interface temperature
- Melt index
- Resin melt point temperature
- Molecular weight of acid
- Resin synthesis feed temperature
- Resin synthesis reactor temperature
- Delta T (reactor temperature—feed temperature).

These property variables were chosen for testing because they can be controlled by either the resin manufacturer or the packaging manufacturer. One purpose of this study was to determine that if the effect of the above variables on hot tack could be quantified, perhaps those variables could be changed to yield any number of desired hot tack values.

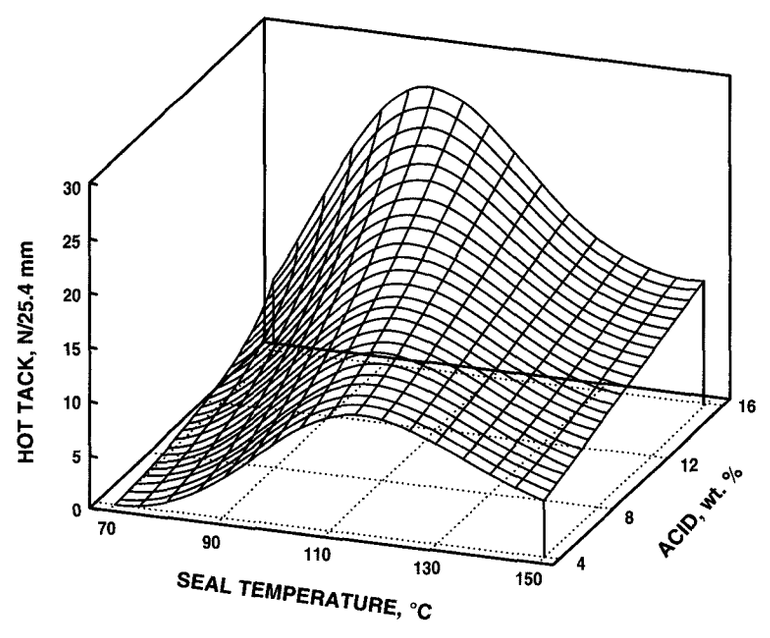
There were several intuitive and theorized reasons that hot tack was believed to be influenced by the above factors. Acid content was tested because a greater acid content was expected to influence the degree of hydrogen bonding in the molten state of the polymer, thereby affecting hot tack results. Seal interface temperature was evaluated because it was expected to affect the viscosity and tackiness of the seal. The melt index was tested to determine if the viscosity of the polymer, as well as melt strength, played a role in hot tack. The resin melt point temperature was evaluated to determine if it affected the temperature at which the peak hot tack values occurred for the different resins. Molecular weight of the acid was tested in an effort to differentiate the results of the methacrylic acid copolymers from the acrylic acid copolymers, and to find any differences in hot tack values between these two classes of copolymers. Finally, the synthesis feed

I. Resins studied

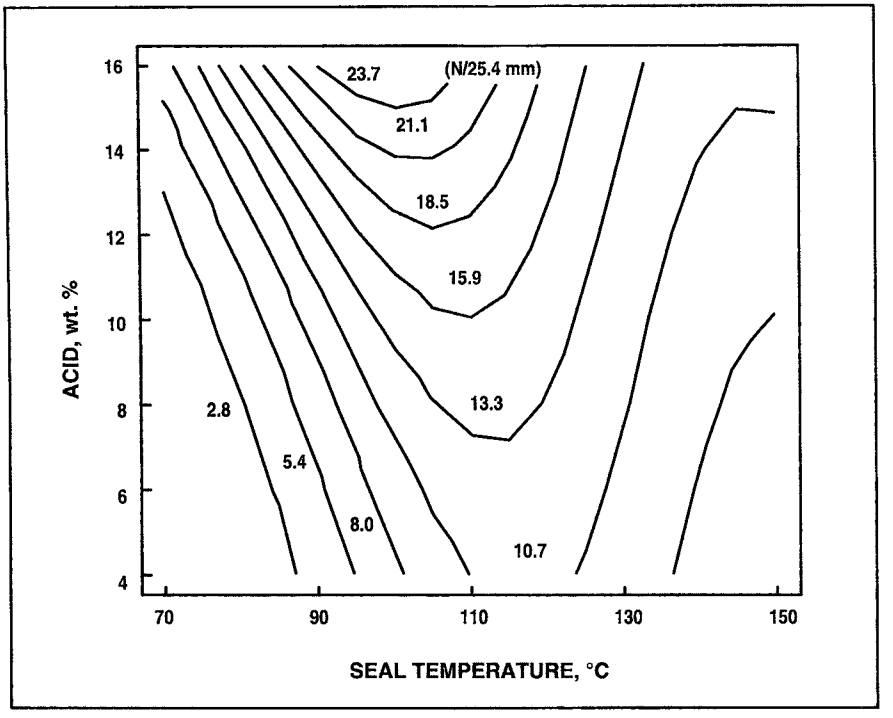
Reference*	MAA, wt.%	AA, wt.%	Melt index, dg/min
Resin #1	4.0		2.5
Resin #2	4.0		7.0
Resin #3	4.0		11.0
Resin #4	6.0		9.0
Resin #5	9.0		1.5
Resin #6	9.0		2.5
Resin #7	9.0		2.5
Resin #8	8.7		10.0
Resin #9	8.7		10.0
Resin #10	12.0		1.5
Resin #11	12.0		7.0
Resin #12	12.0		13.5
Resin #13	15.0		25.0
Resin #14		8.4	3.0
Resin #15		8.4	7.0
Resin #16		11.3	1.3
Resin #17		10.8	10.5

*Resins #1-17 are the following DuPont Nucrel® resin grades in respective order - 0403HC, 0407, 0411HS, 0609HS, 0902HC, 0903, 0903HC, 0910, 0910HS, 1202HC, 1207, 1214, 1525, ARX45-1, ARX48, SH30-2, and 3990.

1. Hot tack as a function of seal temperature and acid content



2. Contour plot of hot tack at different acid levels and seal temperatures



and reactor temperatures, as well as delta T, were evaluated to find possible correlations with the molecular weight distribution or synthesis conditions of the resin with hot tack values.

Experimental procedure

All hot tack values were measured using a DTC hot tack tester, Model 52-E, manufactured by DTC International AB, Sweden. The variable settings of the machine were kept constant at the following values:

- Delay time = 0 s
- Peel speed = 200 mm/s
- Sealing pressure = 0.28 N/mm^2
- Sealing time = 3 s.

The sample-to-sample uniformity of the value of these variables is important because any change may affect the measured hot tack value. Most importantly, the delay time must be maintained as close to zero as possible. Delay time is a measure of the time interval between the opening of the seal jaws and the peeling of the specimen. This value must be minimized because a high delay time would allow the seal interface of the specimen to cool, which would then reflect seal strength rather than hot tack strength of the specimen.

To further minimize inconsistencies due to human error in machine and sample setup, all the samples were prepared and all data were gathered by the same individual.

Sample preparation

All film samples were blown on the same extrusion equipment at a standard throughput of 130 ± 5 g/min, at a 51-μm (2 mil) film thickness. The blowup ratio was standardized at 3:1. Melt temperature and extruder temperature profiles were varied only slightly from one resin to another to get the best resin performance at its particular melt temperature and melt index.

All film samples were backed with ionomer-dispersion-coated paper and joined on a hot pressing machine at a pressure of 55.2 MPa (8000 psi) at 110°C (230°F) for 60 s. In order to be used on the DTC Hot Tack Tester, each sample was uniformly cut to 25.4 mm (1 in.) in width and 330 mm (13 in.) in length. The samples were then inserted into the jaws of the hot tack tester.

The variables manipulated in the experimentation were seal temperature, which was effectively the temperature of the jaws, and the type of film being tested. The temperature was varied in 10-degree increments from 70°C to 150°C. Each value of hot tack used in the statistical regression was the average of three individual determinations on the hot tack tester.

Results

Statistical correlation of data

Each hot tack data value was the average of three determinations. Test results were entered into a computer program called Stratgraphics, by STSC, Inc. Using multiple regression routines, the resin variables listed in the "Variables tested" section were tested to determine if they had any correlation with the hot tack results. After many multiple regression iterations to test different combinations of variables for correlation, two of the variables were found to have a significant correlation with the hot tack data. A combination of acid content and seal temperature explained most of the data, according to the statistical model. This does not mean that the other six variables did not affect hot tack, but simply that their effects were insignificant and could be due to chance alone.

The results of the technique produced the model represented by Eq. 1:

$$HT = \mathrm{EXP}\,(c_1 + c_2T + c_3T^2 + c_4T^3 + c_5A + c_6AT + c_7AT^2) \quad (1)$$

where

HT = hot tack strength, N/25.4 mm

EXP = exponent function (e^x)

T = seal temperature, °C

A = acid content, wt.%

c_1 = –44.167255

c_2 = 1.009

c_3 = –0.007134

c_4 = 0.00001624

c_5 = 1.280342

c_6 = –0.019064

c_7 = 0.00007285.

Quality of model

With the confidence level set at 95%, Eq. 1 had a statistical r^2 value of 0.91, which indicates that the terms in the model explain 91% of the gathered data. The statistical significance value, or P-value, for all the terms in the equation was less than 0.0000, which means that there is a greater than 99.99% chance that there is a real relationship between the terms and hot tack.

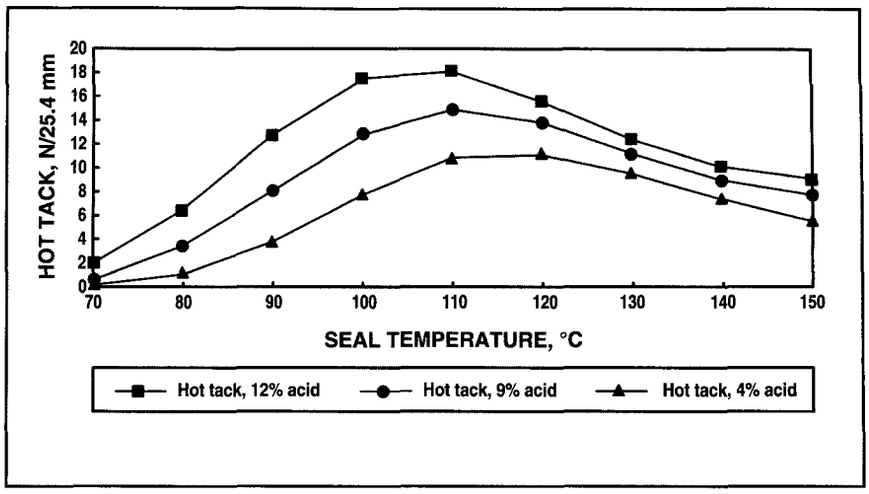
Analysis of model

Equation 1 is a third-order equation with interactions between seal temperature (T), the square of T, and acid content (A). There are two independent variables, T and A, and one dependent variable, HT. This correlation shows that HT is a natural log function. The log function tends to equalize the variability in the lower and higher values of the data points. Because of the wide range of observed hot tack values in this case, the variability in the data had a better correlation with the log of hot tack than with hot tack itself.

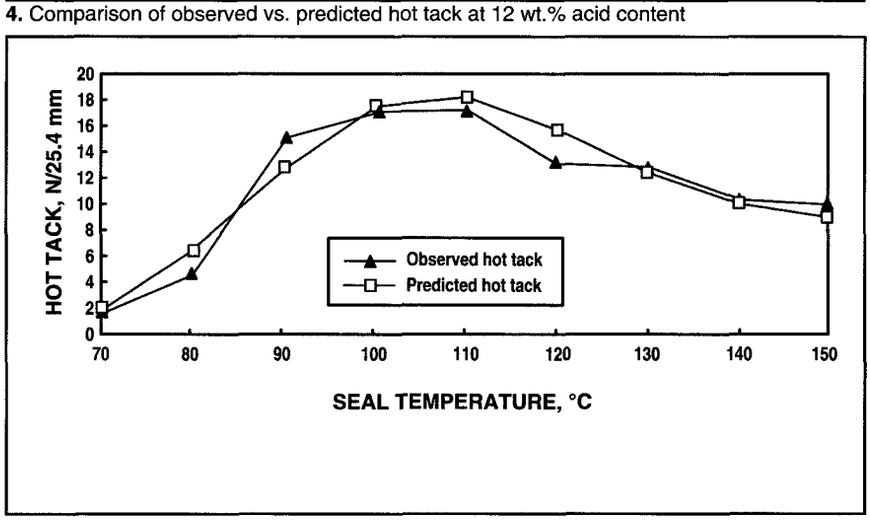
The effect of T is positive and that of T^2 is negative. This implies that hot tack varies parabolically with temperature, with an inflection point at some mid-level temperature. The small but positive effect of the T^3 term raises the hot tack at high temperatures compared to that at low temperatures. The interaction of T and T^2 with A implies that the hot tack behaves differently for different acid levels at any temperature range, and that the effect of acid level on hot tack is not uniform with varying temperatures.

These observations can be visualized in **Fig. 1**, which is Eq. 1 in graphical form. It is a three-dimensional

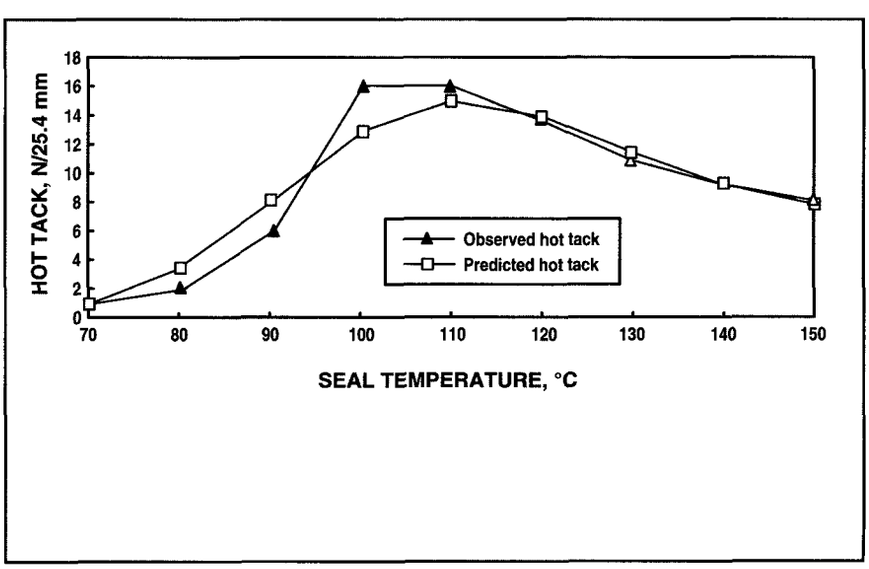
3. Predicted hot tack values at 12%, 9%, and 4% acid levels



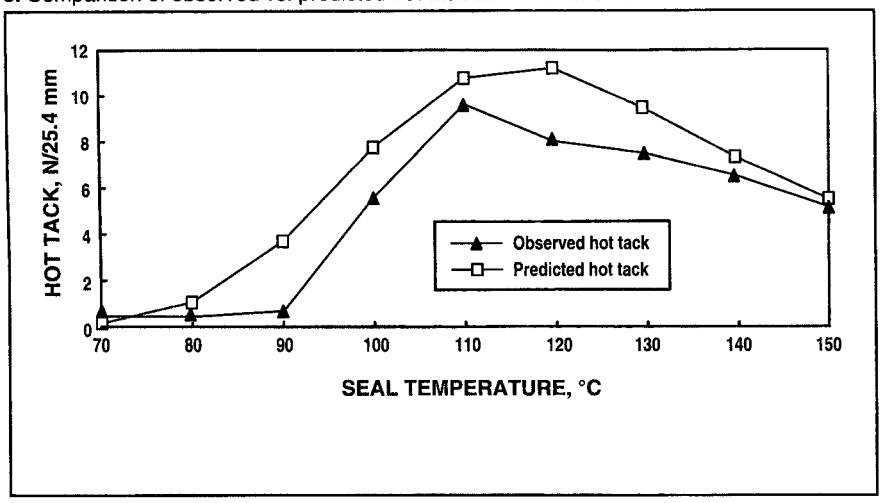
4. Comparison of observed vs. predicted hot tack at 12 wt.% acid content



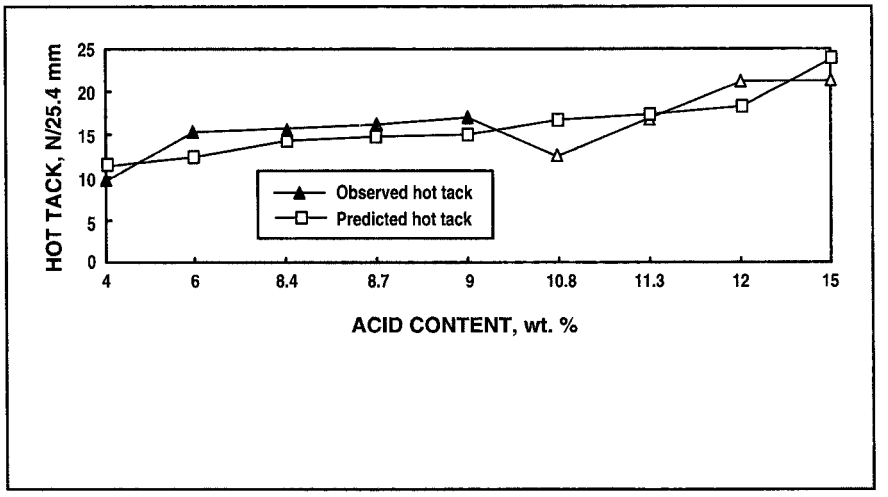
5. Comparison of observed vs. predicted hot tack at 9 wt.% acid content



6. Comparison of observed vs. predicted hot tack at 4 wt.% acid content



7. Comparison of predicted peak hot tack with observed peak hot tack



II. Comparison of predicted and observed values for peak hot tack and lowest temperature for peak hot tack

Acid level, wt.%	*Observed peak hot tack (avg.), N/25.4 mm*	*Observed optimum temp., °C*	*Predicted peak hot tack, N/25.4 mm*	*Predicted optimum Temp., °C*
4.0	9.6	110	11.3	116.3
6.0	15.4	100	12.5	114.4
8.4	15.8	100	14.3	111.6
8.7	16.3	100	14.6	111.2
9.0	17.0	100	14.9	110.8
10.8	12.6	100	16.8	108.2
11.3	16.8	100	17.4	107.4
12.0	21.3	100	18.3	106.3
15.0	21.4	90	23.8	100.9

graph since there are three variables: hot tack, seal temperature, and acid content. Figure 1 illustrates the behavior that was implied by Eq. 1. There is a peak hot tack value at a mid-level temperature for a given acid level. This peak value increases dramatically with acid content. The temperature of peak hot tack strength decreases for higher-acid-content resins.

Note that the peak hot tack strength increases and decreases more sharply at higher acid levels than at lower levels. This implies that, for high-acid-content resins, the hot tack can be raised or lowered dramatically with relatively small changes in seal temperatures.

Figure 1 has been dissected into a two-dimensional contour plot in **Fig. 2** in order to yield more detailed information at different predicted hot tack values. As illustrated in Fig. 2, peak hot tack occurs at lower temperatures for resins with higher acid content. Also, with a given hot tack value, the effective seal temperature range is wider with a higher acid level. Thus, two desirable qualities in films for packaging applications, lower seal temperatures and increased seal temperature range, can be achieved by using higher-acid-content resins.

Figure 3 shows hot tack trend with acid content. It illustrates that hot tack values are greater at higher acid levels at all temperatures for higher-acid-content resins.

Figures 4–6 show the same curves displayed in Fig. 3 along with the curves of corresponding observed values. At 12 wt.% and 9 wt.% acid, the observed and predicted data fit well. At 4 wt.% acid, the observed and predicted data do not correlate as well. This perhaps indicates that the model is less accurate in predicting behavior at the low end of the range of acid levels tested.

Peak hot tack values

Further correlations were made using the same data to more precisely examine some of the implications of these results. In **Fig. 7**, peak hot tack

values (i.e., the highest observed hot tack value at each acid level) have been plotted against predicted peak hot tack values using Eq. 1. Peak hot tack values can be calculated by taking the first derivative of Eq. 1 with respect to temperature and setting the resulting equation equal to zero. After algebraic simplification, this results in Eq. 2:

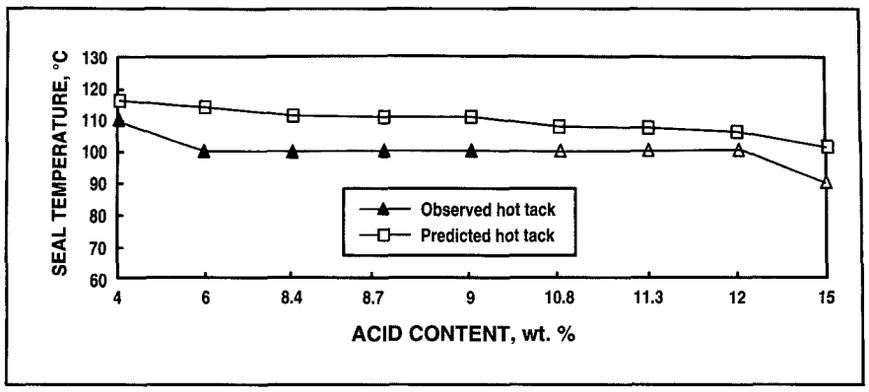
$$0 = c_2 + c_6A + (2c_3 + 2c_7A)T + 3c_4T^2 \quad (2)$$

The maximum value of hot tack would, thus, be the value at which the temperature T and acid content A satisfy Eq. 2 above. By inserting known values of acid contents and calculating the temperature, and then inserting those numbers in Eq. 1, we can predict hot tack peak values. This is presented in **Table II.**

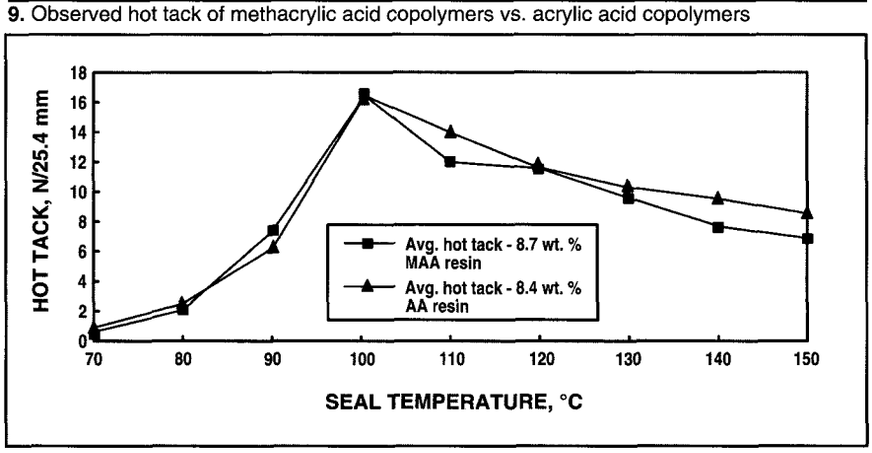
These results have been graphed in **Figs. 7 and** 8. As can be observed in Fig. 7, Eq. 1 is fairly accurate in predicting peak hot tack values at given acid levels. Observed peak hot tack values correspond well with predicted values. However, as shown in Fig. 8, the model is not as accurate in predicting the lowest seal temperature for peak hot tack. In general, the predicted lowest optimum seal temperatures are higher than those observed by approximately 10°C. This 10-degree variability can be partly explained by the fact that the observed data were gathered at temperature increments of 10 degrees. Thus, a 10-degree inaccuracy is possible in the observed data values due to the unknown data points that were not gathered between increments.

Even though the predicted peak temperature is slightly higher, the overall fit of hot tack at the full range of seal temperatures tested is fairly accurate, as illustrated in Figs. 4–6. The model does accurately predict the general trend of lower optimum seal temperature with increasing acid content.

8. Comparison of predicted optimum temperature with observed optimum temperature



9. Observed hot tack of methacrylic acid copolymers vs. acrylic acid copolymers



Methacrylic vs. acrylic acid copolymer resins

In this study, both methacrylic acid (MAA) and acrylic acid (AA) resins with approximately the same acid content (8.7 and 8.4 wt.%, respectively) were tested in order to examine any difference in hot tack behavior. The observed hot tack values are graphed in **Fig. 9.** No significant difference was found between the hot tack performance of acrylic and methacrylic acid copolymers; the peak hot tack was nearly equal in both cases. In addition, the variability, or trend with changes in seal temperature, was nearly equivalent for both types of resins.

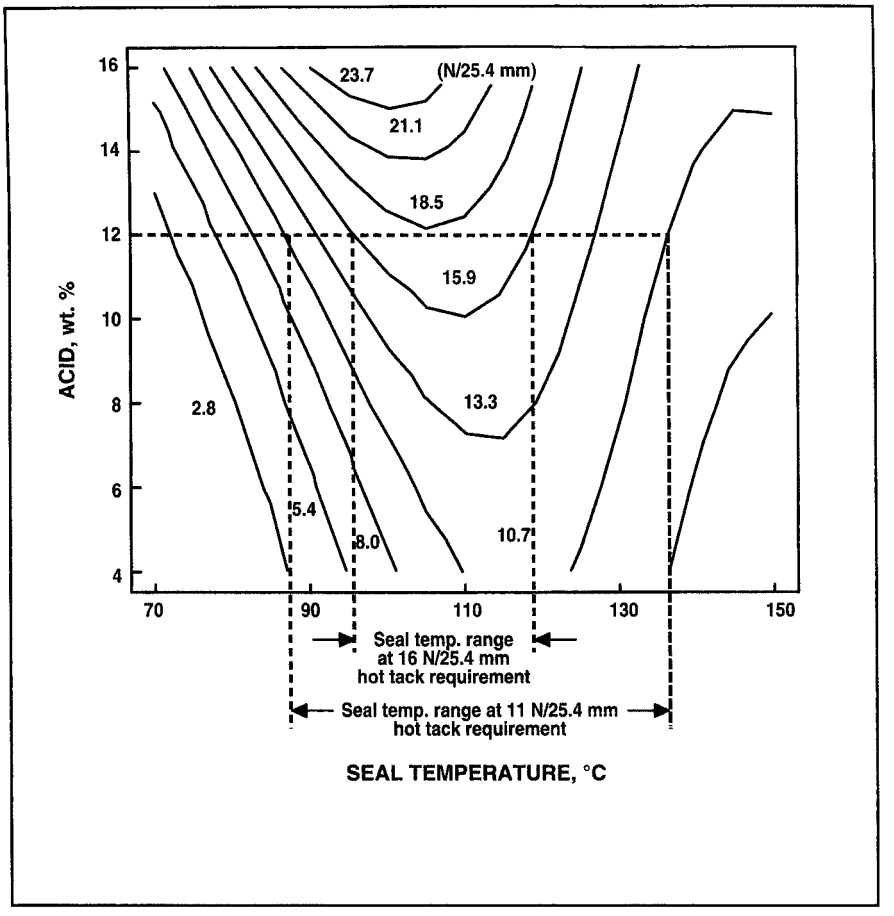
Usefulness of model

This model shows trends and provides insight on the factors that influence hot tack strength. Other than providing a better understanding of hot tack behavior, Eq. 1 and Figs. 1 and 2 may also be used to infer certain information. For example, if given a certain minimum hot tack strength requirement for a package, using Fig. 2, one could find the optimum seal temperature range and the minimum acid content required in the resin. Conversely, if given an operating temperature range constraint, the required acid level and resulting hot tack strength of the resin could be determined.

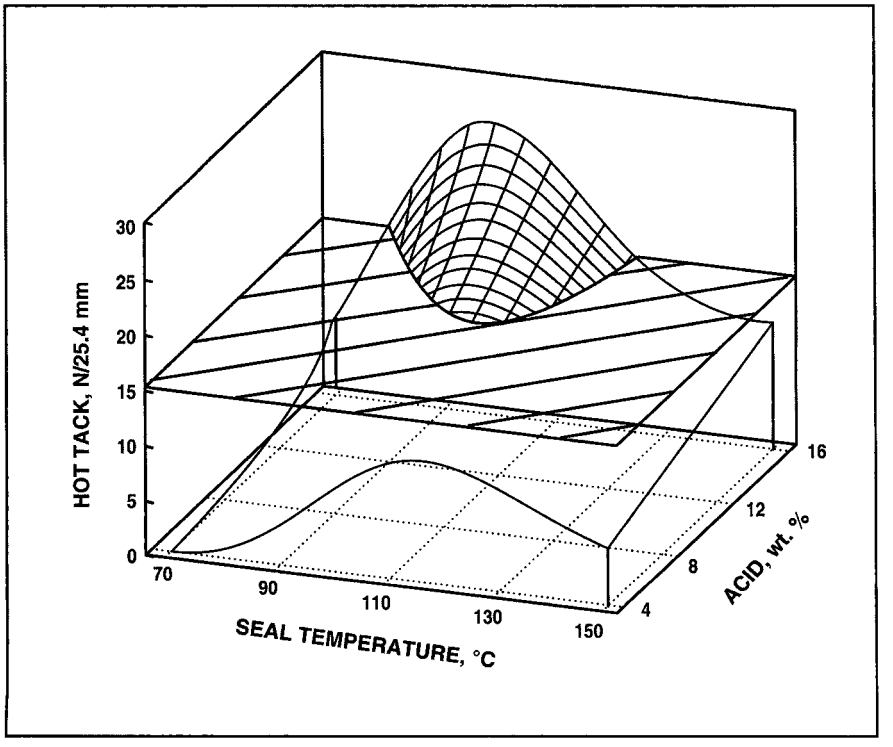
Figure 10 illustrates these techniques for a 12 wt.% acid. At a hot tack requirement of about 16 N/25.4 mm, the predicted seal temperature range will be approximately 96–118°C. At a requirement of about 11 N/25.4 mm, the seal temperature range will be about 87–136°C.

By taking a cross section of Fig. 1, the required "operating surface" could be determined if certain variables are specified. For the case shown in **Fig.**

10. Contour plot of hot tack at different acid levels and seal temperatures



11. Hot tack as a function of seal temperature and acid content



11, a cross section has been taken at a hot tack value of 15 N/25.4 mm. The surface above the cross section illustrates the approximate "surface" of seal temperature range and acid content range that would result if the application required a hot tack of 15 N/25.4 mm.

These graphs are useful for making approximations on hot tack behavior. They are best-fit models based on tested data and should not be used to predict exact behavior. Any information gathered from the graphs should be confirmed by testing under desired conditions.

Summary and conclusions

Acrylic as well as methacrylic acid copolymers with a wide range of acid content and melt indices were tested for hot tack strength at different seal temperatures. These hot tack values were statistically evaluated for correlation with eight resin variables. The results indicate that the major factors influencing hot tack strength are the acid content of the resin and the temperature of the seal. According to the statistical model, these two factors and their interactions explain 91% of the observed data, with a 99.99% chance that the correlations are real.

The model predicts that hot tack strength increases with acid content and has peak values at seal temperatures between 100°C and 116°C for the resins tested (see Figs. 1 and 2). Melt index, melt temperature, type of acid, and resin synthesis feed and reactor temperatures have an insignificant effect on hot tack. In addition, no significant difference was found between hot tack performance of acrylic and methacrylic acid copolymers (see Fig. 9).

The model was fairly accurate in predicting the value of peak hot tack strength at a given acid level and showing its trend with seal temperature. However, it was not as accurate in predicting the lowest temperature at which the peak hot tack values would occur (see Fig. 8).

The significance of this model is not only the two factors it shows affecting hot tack, but also the factors that have an insignificant effect on hot tack. The correlation is illustrated in the form of an equation (Eq. 1) and a three-dimensional graph (Fig. 1). This study allowed us to gain a better understanding of an important phenomenon in packaging applications, and make more informed decisions when confronted with hot tack issues. TJ

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