JEAS

JOURNAL OF ENGINEERING

AND

Journal of Engineering and Applied Sciences, Volume 18, Number 1, June 2021, 366-380

# Influence of Interfacial Parameters on the Adhesion of Aluminium on Mild Steel using Pressure Sensitive Adhesives

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**Abstract**
The influence of interfacial parameters on the adhesion of pressure sensitive adhesives was investigated. Study on effect of adhesive rheology, interface parameters, surface modification and adhesion strength prediction from correlation between fracture energy/ thermodynamic work of adhesion was done. Research analysis was carried out using concept of physical and practical adhesion approaches. Test liquids Ethylene glycol, Glycerol and Ethanol were used to derive contact angle. Contact angles snapshots for both substrates and adhesives were done using high definition Nikon D80 camera. Low Bond Axisymmetric Drop Shape Analysis with Image J software was used to measure contact angle values. Fowkes law was used to calculate surface free energy for both adhesives and substrates. Owendts-Wendt law was employed to calculate thermodynamic work of adhesion. Surface roughness of substrate was evaluated using SRT-6100 surface roughness tester. Characterization of the substrates was done with XMET 700XRF spectrometer. Analytical tools such as SPSS, Excel, Minitab 3D surface plot plot were used for data analysis. Correlation evaluation for fracture energy and thermodynamic work of adhesion showed an inverse relation measured across adhesives used. As follows, Epoxy (-0.387), Natural rubber (- 0.6), Acrylic (-0.345), Silicon (-0336). 3D Surface Plot showed linear, inverse and partial relation for polar and dispersive component of substrates and adhesives. A novel way of evaluating adhesion strength without breaking of bond was put forward. This involves deriving the Adhesion Ratio across the bonds. Additionally, interfacial attraction between substrates and adhesives along the lines of dispersive and polar components of surface free energy were used to evaluate adhesion strength. Hence, this work established that when substrate and pressure sensitive adhesives of opposite polarities are bonded at interface, stronger work of adhesion is achieved. This extends the hypothesis that close ratio matching of polarity components between adhesive and substrates predicts stronger adhesion.

**Keywords:** Interfacial Parameters; Adhesion; Adhesive rheology; Energy; Surface Energy; Contact Angle

## 1. Introduction

The basic demand is that emergency signs (made from aluminum plate) be pasted on the vertical wall surface as a means to alert staff of the presence of first aid safety equipment for use in case of emergency situation such as inferno. The challenge to executing this task is that use of fasteners such as nails, pins and boring the surfaces will lead to destruction of surface aesthetics, increases stress concentration at the point where fasteners are used and adds to the weight the surfaces bear. However, adhesive offers alternative to use of fasteners and the above enumerated challenges are eased off, but upon the application of the adhesives to stick the sign on the vertical wall surfaces most failed to paste the signs. The major suspicion is that the failure of the signs to stick to the surface was a result of failure of interfacial parameter to allow adherence between the surfaces (Kim et al 2010). Remarkably the research employs the use of work of adhesion and fracture energy to find out the reason for the failure (Wei and Yueguang 2012). The work of adhesion measures the relation between interfacial energy between adhesives and substrates (Rudenauer, 2013) and the fracture energy is fundamental to the process of delamination (Mokhtari et al, 2017). The delamination is as a result of debond across two materials which stop adhering to each other.

Available literature shows that the quality and strength of bond is only measurable upon destruction of the bond (Ashley et al 2014, Gerald et al 2013). The bond destruction is hinged on measuring the fracture energy. (Giurgiutiu et al 2001, Howson 2011) demonstrated a relationship between the interfacial energy and fracture energy for asphalt-blinder and it's aggregate. Phattanarudee (1998) observed that thermodynamic work of adhesion in presence of moisture predicts decrease in adhesion for lead frame/ adhesive interface. Danielle et al (2018), observed a good relationship between the work of adhesion and adhesion strength from peel strength.

Thus, this study seeks to explore the prediction of the adhesive bond/adhesive strength without destruction, by utilizing the thermodynamics work of adhesion. The thermodynamic work of adhesion is obtained by the measure of physical adhesion, while the fracture energy is derived by the measure of practical adhesion. Each of the variables (fracture energy or the thermodynamic work of adhesion) gives its own measurement. The result of the measurement obtained from the thermodynamic work of adhesion is tested for a correlation with the measurement obtained from the fracture energy. The measurements are done with the aid of interfacial parameters, and these parameters help to achieve expectations in the derived direction.

## 2.0 Materials and methods

### 2.1 Materials.

The materials used in this research are; substrates (flat aluminium and mild steel plate), probeliquids **(**ethylene glycol, glycerol and ethanol), surface cleaning liquid (petroleum ether), camera (Nikon D80), syringes**,** low bond axisymmetric drop shape analysis software (LSADSA), pressure sensitive adhesives (Abro- 200, RTV Silicon sealant, abro epoxy steel, kwik-set consists of an epoxy resin and hardener; AB-Adhesive Ever-King multipurpose acrylic adhesive; cow skin animal based pressure sensitive adhesive and general purpose natural rubber based pressure sensitive adhesive), Universal Tensile Testomeric Machine**,** XRF Spectrometer, and Surface Roughness Tester.

### 2.2 Methods

#### 2.2.1 Substrate Preparation and Characterization

The substrate sample used in the research had flat surface and was cleaned with cotton wool soaked in PET to clean the surface from dust, grease and were dry at room temperature, to ensure that the PET does not mix with the test liquids. The characterization of the substrate was done with an XRF-Spectrometer. The XRF- Spectrometer is placed in contact of each substrate for at least 15 seconds. The machine thus gives information on the chemical composition of each substrate.

#### 2.2.2 Pressure Sensitive Adhesive Film Preparation and Characterization

The natural rubber base pressure sensitive adhesive used for the experiment was specially designed for research using the following specification: chlorinated natural rubber = 1000gm; phenolic resin = 250gm; and light magnesium carbonate = 125gm. The solvents used are: toluene = 1000ml; acetone = 300ml; and premium motor spirit = 150ml.

The sample prepared was divided into three samples; A, B, and C.

**Sample A:**1000gram of chlorinated natural rubber was added to 150ml of acetone, and 500ml of toluene. The mixture is well agitated till there is complete dissolution of the components.

**Sample B:**250gramof phenol resin was differently mixed with balance of 150ml of acetone and 500ml of toluene. The mixture is well agitated till complete dissolution of the components. The mixture (phenol resin, acetone and toluene) was further treated with 125gram of light magnesium carbonate. The resulting mixture was the agitated, till complete dissolution was achieved.

**Sample C:** Samples A and B were mixed together and agitated to ensure complete dissolution. Afterwards 150ml of premium motor spirit was added to the mixture of sample A and B to give sample C. The new sample C was as well agitated and the mixture (sample) was stored in a tight container ready for use.

#### 2.2.3Contact Angle Measurements

Flat substrate surfaces were mounted on the block, while the adhesive film was spread on a flat glass and mounted on the block. The light was to illuminate the place in order to obtain a bright image of the droplet. The syringe was brought close to the flat substrate to release test liquid drops to the substrate/film surface. The frozen drop images of the side views of the test liquid were taken by the high definition camera. The snap shots were taken at a rate of 10 frames per second, while the distance between the syringe needle and substrate was reduced. This is to minimize the effect of air current distortion of the drops. The contact angle was measured 7 times for each substrate, with the average and standard derivation calculated.

### 2.2.4 Determination of Surface Energy

The determination of the surface energy of either substrate or adhesive film is dependent on the contact angle. The average values of the contact angle measured were used to calculate the polar and dispersive component of the surface free energy.
The surface energy is calculated by the use of equation 1. (Flinn and Ashley, 2010)

$$\frac{\gamma_{lv}(\cos\theta+1)}{2\sqrt{\gamma_{lv}^{p}}} = \sqrt{\gamma_{sv}^{d}}\left[\sqrt{\frac{\gamma_{lv}^{d}}{\gamma_{lv}^{p}}}\right] + \sqrt{\gamma_{sv}^{p}} \quad (1)$$

Where the variables are represented as follows:

$\gamma_{lv}$ total surface energy between the liquid and the vapour
$\gamma_{lv}^{p}$ polar component of the surface energy between the liquid and vapour.
$\gamma_{lv}^{d}$ dispersive component of the surface energy between the liquid and the vapour.
$\gamma_{sv}^{p}$ polar component of the surface between the solid and the vapour.
$\gamma_{sv}^{d}$ dispersive component of the surface energy between the solid and vapour.

The Kaelble plot for each test liquid was plotted. The Kaelble plot shows the relationship between the contact angle measurements gotten from each test liquid. To this effect, equation 2 was plotted as Y coordinate while equation 3 was plotted as X- coordinate. The polar component of the surface energy was calculated as the Y-intercept of the plot squared. The dispersive component of the surface energy was the slope of the plot squared. The surface energy of the test liquid components were derived from the literature ACC Dyne Test Manual. However the surface free tension of each adhesive film or substrate was calculated by the summation of the surface free energy of components.

$$\gamma_{sv} = \gamma^{d} + \gamma^{p} \quad (2)$$

### 2.2.5 Determination of the Interfacial Surface Energy

The interfacial energy exists across two boundaries. The first is between the pressure sensitive adhesive and aluminum. Second is between the pressure sensitive adhesive and substrate. Fowke's law is used to derive the relation as follows;

$$\gamma_{sv} = \gamma_{sv} + \gamma_{sv} - 2\left(\gamma_{s}^{d}\gamma_{l}^{d} + \gamma_{s}^{p}\gamma_{l}^{p}\right) \quad (3)$$

Applying the Owendts Wendt law to obtain the interfacial surface energy is represented as follows:

$$\gamma_{sl} = \gamma_{sv} + \gamma_{lv} - 2\left(\gamma_{sv}^{d}\gamma_{lv}^{d}\right) - 2\left(\gamma_{sv}^{p}\gamma_{lv}^{p}\right)^{1/2} \quad (4)$$

Where equation 3.6.is further simplified with regards to the contact angle as follows:

$$\gamma_{sv}(1 + cos\theta) = 2\left(\gamma_{sv}^{d}\gamma_{lv}^{d}\right) + 2\left(\gamma_{sv}^{p}\gamma_{lv}^{p}\right)^{1/2} \quad (5)$$

### 2.2.6 Determination of Thermodynamic Work of Adhesion

The thermodynamic work of adhesive refers to the fundamental adhesion. It deals with the forces acting among atoms across an interface. The thermodynamic work of adhesion shall be calculated across boundaries. The boundaries are between the pressure sensitive adhesive and aluminum or pressure sensitive adhesive and substrate surface. This stipulated as follows;

$$W_{a} = \gamma_{sv} + \gamma_{lv} - \gamma_{sl} \equiv \gamma_{lv}(1 + cos\theta) \quad (6)$$

### 2.2.7 Determination of Fracture Energy

Fracture energy was measured using the "Pull-off test". The pull-off test regards the amount of displacement generated by an applied force. It shows the stress-strain relation of each particular adhesive for each bond pair. The bond pair represents the interaction among the Aluminum/Pressure Sensitive Adhesive/ Substrates and the interfaces across each joint. The various adhesives bonds, the different surfaces with the Aluminum (emergency sign) plate. The pull-off test was conducted with the aid of a Testomeric Universal testing machine, Model M500- 25CT, computerized and 27KN capacity. The type of tensile test conducted was PWG25W and the test conducted at test speed of 50millimeters/minute (50mm/min.).

### 2.2.8 Determination of the Surface Finish of the substrates.

The substrates surface roughness were measured with the aid of Surface Roughness Tester Model No. SRT-6100, 4 digits, 10mm, LCD with blue light. It has a measurement range Ra 0.05-10.00 µm, Rz 0.1-50.0µm.

### 2.2.9 Prediction of Adhesion through non-destructive method

The adhesion at each bond type can be predicted using the combination of interfacial parameters vital to enforce tack. These include thermodynamic work of adhesion, work of cohesion, work of spreading, total work of adhesion. The combination of these parameters is used to derive adhesion ratio. The adhesion ratio is used to measure the bond performance and quality across each bond type. The force at peak derived from the tensile test is established as the tack from practical adhesion test (fracture energy). The value from adhesion ratio of each bond type was found to be in the range of -1 to +1 for each bond type. The following equations express how the interfacial parameters such as adhesion, cohesion and spread were used to derive the adhesion ratio.

Thermodynamic work of adhesion $W_A$

$$W_A = \gamma_{sv} + \gamma_{lv} - \gamma_{sl} \quad (7)$$

Work of cohesion

$$W_A = 2\gamma_{lv} \quad (8)$$

Work of spreading

$$W_S = W_A - W_C \quad (9)$$

Total work of adhesion at bond $W_{AC}$

$$W_{AC} = W_A + W_C \quad (10)$$

Adhesion Ratio is ratio of work of spread to Total work of adhesion and represented as A.R.

A.R. = $W_S / W_{AC}$

## 3.0 Results and Discussion

### 3.1 Analysis of relationships: between dispersive and polar components of surface free energy for substrates and pressure sensitive adhesives, thermodynamic wok of adhesion and fracture energy across different bond types.

Energy is consumed during the breakage of a bond. The thermodynamic work of adhesion measures the theoretical or physical adhesion. The fracture energy measures the practical adhesion. Energy consumed during the breaking of bond (fracture) was lost due to dissipation and viscoelastic deformation. The electrostatic model adhesion assumes that during adhesion, either the substrate or the adhesive is positive, while the other is negative. This model is particularly applicable where fracture or debond has taken place across a bond. Thus, the inter-molecular forces of dispersive and polar forms built around dipoles-dipoles, Vander Waals and acid-base interaction exist at the adhesion interface. These forces play major roles in defining the adhesion behavior at any interface.

### 3.2 Analysis of the relationship between Thermodynamic work of Adhesion and Fracture Energy

Table 1 expresses the value of thermodynamic work of adhesion and fracture energy for each bond type. The bond type consists of the substrate and accompanying bond pairs. The bond pair is a collection of interfacial relation between each pressure sensitive adhesive and aluminium (the sign board). However, the significant difference in the value of the two interfacial parameters can be linked to observations made at tensile test. During tensile test there is a significant amount of viscoelastic energy generated due to deformation of pressure sensitive adhesive layers. The viscoelastic energy, though cannot be quantified by this research work consist of the thermodynamic work of adhesion and viscoelastic energy. Analytical tools such as Minitab 2018, Microsoft Excel, Statistical Package for Social Science, were employed to analyze data derived from the research.

**Table 1. Thermodynamic Work of Adhesion (TWA) and Fracture Energy (FE) for each Bond type on Mild Steel**

| Acrylic/Aluminum | | Epoxy/ Aluminum | | Natural rubber/ Aluminum | | Silicon/ Aluminum | | Cow skin/ Aluminum | |
|---|---|---|---|---|---|---|---|---|---|
| FE (Nm) | TWA ($J/m^2$) | FE (Nm) | TWA ($J/m^2$) | FE (Nm) | TWA ($J/m^2$) | FE (Nm) | TWA ($J/m^2$) | FE (Nm) | TWA ($J/m^2$) |
| 36.430 | 68.150 | 7.217 | 92.580 | 53.917 | 130.600 | 52.712 | 96.11 | 59.545 | -28.330 |

**3.2.1 Statistical Package for Social Science (SPSS)**

Statistical Package for Social Science (SPSS) was used to investigate the relationship between the two quantities (thermodynamic work of adhesion and fracture energy). Table 2 shows the result of the test.

**Table 2: Result of SPSS Pearson correlation between Thermodynamic Work of Adhesion (TWA) and Fracture Energy across each bond formed by each Pressure Sensitive Adhesive**

| Adhesives | Statistics | TWA |
|---|---|---|
| **Natural rubber** | Pearson Correlation (r) | -0.604 |
| | P value | 0.396 |
| **Epoxy** | Pearson Correlation (r) | -0.387 |
| | P value | 0.613 |
| **Acrylic** | Pearson Correlation (r) | -0.345 |
| | P value | 0.655 |
| **Silicon** | Pearson Correlation (r) | -0.336 |
| | P value | 0.664 |
| **Cow skin** | Pearson Correlation (r) | -0.544 |
| | P value | 0.456 |

The measurements were carried out according to each pressure sensitive adhesive used for the research. Each bond type/pressure sensitive adhesive is accompanied by a corresponding value of thermodynamic work of adhesion, significant correlation and Pearson correlation.

The significance correlation which was described as non-existing, ($P>0.05$) indicates that there is no direct relationship between the two variables. The variables referred here are the thermodynamic work of adhesion and the fracture energy. However this judgment is not feasible because SPSS significant correlation is based on sample size. The larger the sample size the possibility of achieving a significance correlation were $P>0.05$, which is perceived as acceptable. Thus the research ignores the preconception of significance of correlation and concentrates on the Pearson correlation. This is because the sample size 'N' is just 30 thus limited by SPSS standard. Negative Pearson correlation implies that both variables (fracture energy/ thermodynamic work of adhesion) interchanged roles. The Pearson correlation for Natural rubber shows a value of -0.604. This means that a strong correlation exists though negative. The implication is that as the fracture energy across bonds held by natural rubber adhesive rises, the thermodynamic work diminished by a factor of 0.604 or 60%. Likewise the fracture energy diminishes by a factor of 0.604 or 60% and the thermodynamic work of adhesion for epoxy pressure sensitive adhesive. The Pearson correlation value is -0.387. This is moderate, though negative. It indicates that the fracture energy rises while the thermodynamic work of adhesion diminished by a factor of 0.387 or 38.7% and vice versa. For acrylic pressure sensitive adhesive, the Pearson correlation value is -0.345. This is moderate, though negative. It indicates that the fracture energy rises while the thermodynamic work of adhesion diminished by a factor of 34.5% or 0.345 and vice versa. For silicon pressure sensitive adhesive, the Pearson correlation value is -0.336. This is moderate, though negative. It indicates that the fracture energy rises while the thermodynamic work of adhesion diminished by a factor of 0.336 or 33.6% and vice versa. For cow skin pressure sensitive adhesive, the Pearson correlation value is -0.544. This is moderate, though negative. It indicates that the fracture energy rises while the thermodynamic work of adhesion diminished by a factor of 0.544 or 54% and vice versa.

**3.2.2 Microsoft Excel Software 2010**

This was used to investigate the relationship between the Thermodynamic work of adhesion and Fracture Energy. The result showed that for bond types involving natural rubber pressure sensitive adhesive. The correlation is -0.66796. This is considered high, though negative. The indication shows that while the fracture energy rises the thermodynamic work of adhesion diminished by a factor of 0.66796 or 66.79% and vice versa. Bond type involving epoxy pressure sensitive adhesive, showed a correlation of -0.38772. This is moderate. It shows that the fracture energy rises, while the thermodynamic work of adhesion diminished by a factor of 0.38772 or 38.77% and vice versa. Bond type involving acrylic pressure sensitive adhesive, showed a correlation of -0.3447. This is moderate. It shows that the fracture energy rises, while the thermodynamic work of adhesion diminished by a factor of 0.3447 or 34.47% and vice versa. Bond type involving silicon pressure sensitive adhesive, showed a correlation of 0.3491. This is moderate. It shows that the fracture energy rises, while the thermodynamic work of adhesion diminished by a factor of 0.3491 or 34.91% and vice versa. Bond type involving cow skin pressure sensitive adhesive, showed a correlation of -0.5444. This is moderate. It shows that the fracture energy rises, while the thermodynamic work of adhesion diminished by a factor of 0.5444 or 54.44% and vice versa.

**3.3 Analysis of the Relationship between Dispersive and Polar Component of the Surface Energy for both Substrates and Pressure Sensitive Adhesive**

The relationship between the polar component and dispersive components of the surface free energy for both substrates and pressure sensitive adhesive, were used to derive surface free energy for both materials. This was done by the use and implementation of scatter plot of the Fowkes equation on both materials. These relationships were analyzed as follows:

**3.3.1 Microsoft Excel**

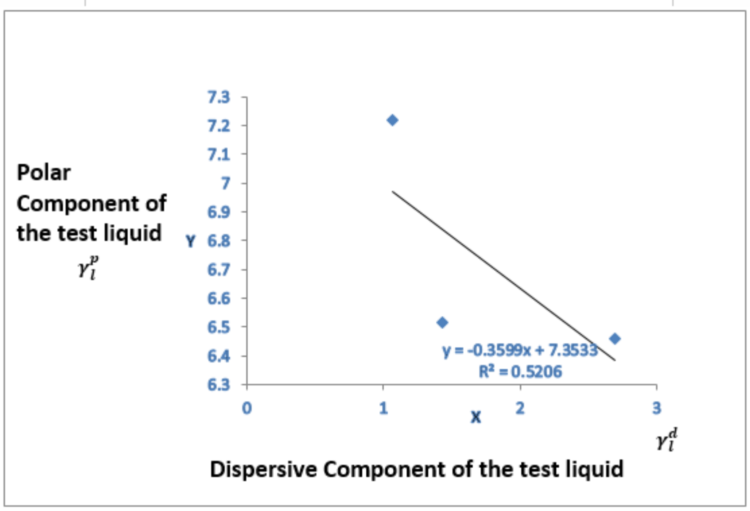


**Figure 1: Scatter Plot of Fowkes Equation for Cow Skin Pressure Sensitive Adhesive to Derive Surface Free Energy for the Adhesive**

Fig. 1 shows a scatter plot. This scatter plot was obtained by plotting the polar and dispersive component of the 3 test liquids using the Fowkes equation. The graphical analysis resulted in a regression equation $Y = -0.3599x + 7.3533$. The sum of the squares of the intercept and slope was used to determine the surface free energy of Cow skin pressure sensitive adhesive. The coefficient of determination $R^2$= 0.5206 or 52.1%. This regression analysis shows that the dependent variable Y representing the surface free energy of cow skin adhesive can be predicted by the result of the values of the polar and dispersive component (independent variables). It shows a strong relationship between the values of the surface free energy for the cow skin pressure sensitive adhesive and its component (adhesive and polar) surface free energy good regression.

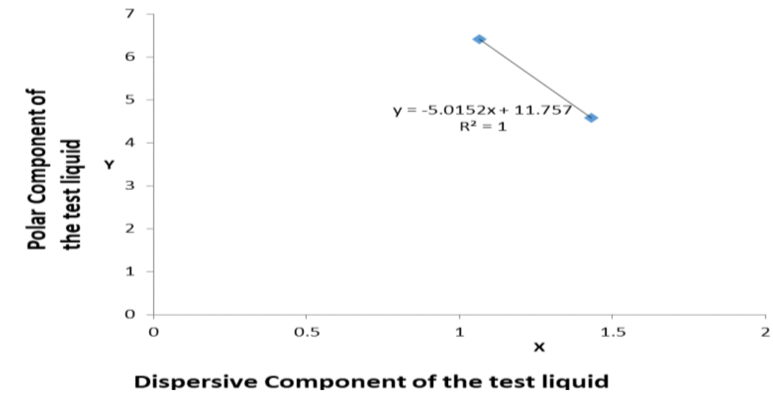


**Figure 2: Scatter Plot of Fowkes Equation for Acrylic Pressure Sensitive Adhesive to Derive Surface Free Energy for the Adhesive**

Figure 2 is a scatter plot representing the relationship between polar and dispersive components of acrylic pressure sensitive adhesive. The regression equation is as follows: $y = -5.012x + 11.757$. The sum of the squares of both the intercept and slope give the surface free energy of acrylic adhesive. The regression value is 1. This suggests harmony in the role of dispersive and polar components respectively in determining the value of the surface free energy for the adhesive. However the value of the regression must have been affected by the use of only two test liquids (ethylene glycol and glycerol) for the analysis. The third test liquid ethanol exhibited spontaneous wetting when dropped on the film of the acrylic. This stopped the image of the contact angle from being derived at snapshot and subsequent calculations were limited. Good regression.

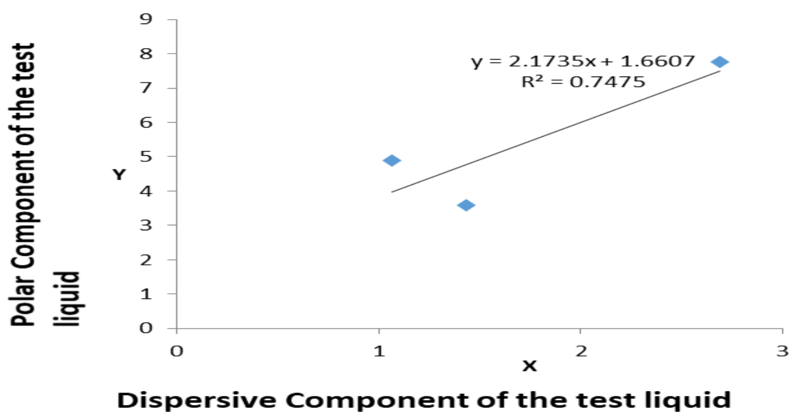


**Figure. 3: Scatter Plot of Fowkes Equation for Silicon Pressure Sensitive Adhesive to Derive Surface Free Energy for the Adhesive**

Figure 3 is a scatter plot representing the relationship between polar and dispersive components of silicone pressure sensitive adhesive, with a regression equation of $y = 2.1735x + 1.6607$. The sum of the squares of both the slope and intercept gives the surface energy of the silicone adhesive. The coefficient determination is 0.7475 or 74.75%. It indicates a strong relationship between the values of the dependent variable the surface free energy and the independent variables the polar and dispersive component. This is a good regression.

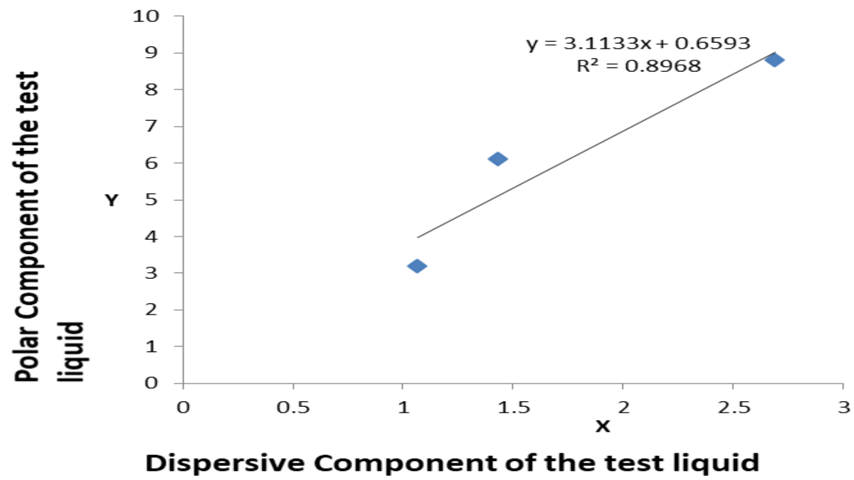


**Fig. 4: Scatter Plot of Fowkes Equation for Epoxy Pressure Sensitive Adhesive to Derive Surface Free Energy for the Adhesive**

Fig 4 is a scatter plot represents the relationship between the polar and dispersive component of epoxy pressure sensitive adhesive. The regression equation is as follows: $y = 3.1133x + 0.6593$. The sum of the squares of the slope and intercept give the surface free energy of the epoxy. The value of the coefficient of determination is 0.8968 or 89.68%. The regression shows a strong relationship between the dependent variable (surface free energy) and independent variable (polar and dispersive component). Thus the independent variable can predict the dependent variable by 89.682, good regression.

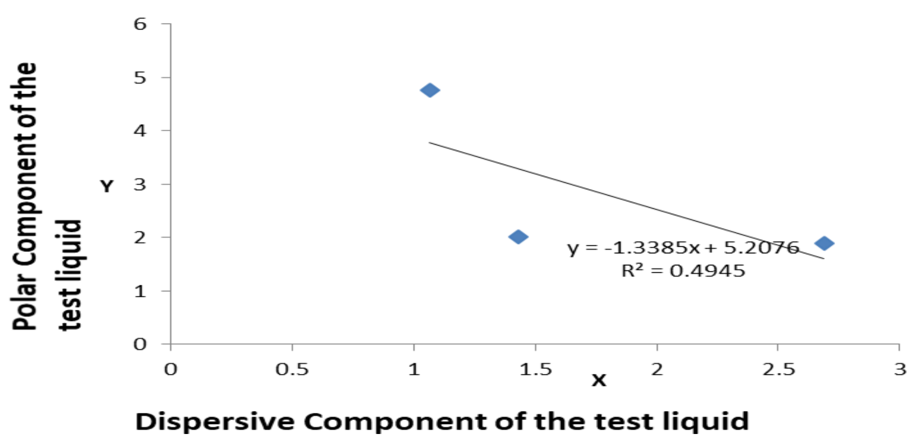


**Fig. 5: Scatter Plot of Fowkes Equation for Mild Steel Substrate to Derive Surface Free Energy for the Substrate**

Fig 5 is a scatter plot representing the relationship between the polar and dispersive component of Mild steel. The linear regression is as follows: $y = -1.3385x + 5.2076$ and $R^2$=0.4945 (coefficient of determination). The sum of the square of both slope and intercept of the regression gives the surface free energy of the Mild steel. The value of the coefficient of determination of 0.4945 is a result of 49.45%. The result gives a moderate regression. Thus the value of the polar and dispersive component predicts the value of the surface free energy by 49.45%.

**Table 3: Results of SPSS Correlation and Regression Analysis between Dispersive components and Polar components of Surface Free Energy for both substrates and Pressure Sensitive Adhesives.**

| | R | $R^2$ | B | T | P value |
|---|---|---|---|---|---|
| Mild steel | 0.703 | 0.494 | -1.338 | -0.989 | 0.504 |
| Cow skin | 0.722 | 0.521 | -0.360 | -1.042 | 0.487 |
| Natural rubber | 0.904 | 0.817 | 3.898 | 2.115 | 0.281 |
| *Acrylic | - | - | - | - | - |
| Silicon | 0.865 | 0.748 | 2.173 | 1.721 | 0.335 |
| Epoxy | 0.947 | 0.897 | 3.113 | 2.948 | 0.208 |

From table 3, the correlation coefficient (R) above 0.7 indicates that a very strong linear positive relationship between disperse and polar components. The coefficients of determination ($R^2$) were above 0.5 indicating that over 50% of the variation in polar component can be attributed to disperse component. In Table 4.10, the significance relationship was described as $P>0.05$. The verbal interpretation is that the relationship between the variables was not feasible. However, this judgment was considered null in the context of this research. This is because validity of significance of relationship which is expected to be $P< 0.05$ is applicable when the number of variable is at least 200 in number. The number 200 is just sample expected to be picked from major population of variables. This research work has a total of 30 samples formed by the drops of each test liquid on each of the substrate and five pressure sensitive adhesive respectively. The acrylic adhesive generated results from two test liquids, ethylene glycol and glycerol while ethanol produced a spontaneous wetting on acrylic sample. The rubber tile substrate generated results from two test liquids ethanol and glycerol while ethylene glycol produced a spontaneous wetting on rubber tile sample. This development limited the analysis of the sample. Table 3 shows that data for both dispersive and polar components for both substrates and pressure sensitive adhesive generated a correlation coefficient above 0.7. This means that there is a highly valid relationship between components of surface free energy for both pressure sensitive adhesive and substrates. The correlation helps to compare the ratio between the dispersive and polar components of the surface free energy for either substrates or adhesives. This comparison helps to predict adhesion between substrate and pressure sensitive adhesive. This is because when the ratio between the dispersive and polar component of the surface free energy the adhesives and substrates are known. It can help to predict adhesion between the two materials. The coefficient of determination across the polar and dispersive components for both substrates and pressure sensitive adhesives yield a value of above 0.5. It indicated that 50% of the variation in the polar component of either the substrate or pressure sensitive adhesive can be attributed to the dispersive component in the both materials. This means that for a substrate or pressure sensitive adhesive to be designated as a polar material, that the outcome of such verdict is influenced by the dispersive component of the surface free energy of such material. For this research work, it can be adduced that the amount of influence dispensed by the dispersive component on the polar component of the free energy is at 50% magnitude. Invariably, it can be stated that the dispersive forces in this research work also influenced the outcome of interfacial energy, wetting, adhesion amongst Substrate and Pressure Sensitive Adhesives. This is because it affects the role the polar component will play in the interfacial interactions.

### 3.3.2 3D-Surface Plots (Minitab 3D Surface Plots)

The 3D-Surface Plots shows the relationship between surface free energy and the polar components as well as the dispersive components of the surface free energy for both substrates and adhesives. The surface energy is represented as the Z axis. The Polar component of the surface free energy is represented as Y axis. The Dispersive component of the surface free energy is represented as the X axis.

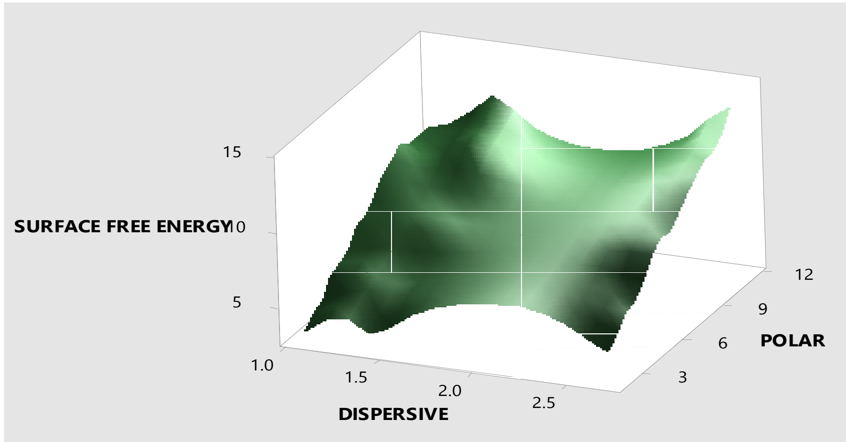


**Fig 7: 3D Surface Plot of Surface Free energy vs Polar and Dispersive Surface Free Energy for Cow Skin**

Fig 7 shows a parabola for the relationship of the dispersive free energy component values with regards to corresponding values with the surface free energy. The polar free energy components maintain a linear relationship with a corresponding with a corresponding rise in the values of the surface free energy. The surface free energy of cow skin pressure sensitive adhesive relationship can be expressed as a quadratic function, while the polar is a linear relationship.

This to obtain the value of the surface free energy for the cow skin pressure sensitive adhesive, the contribution of the polar component is a square of the contribution of the dispersive components.

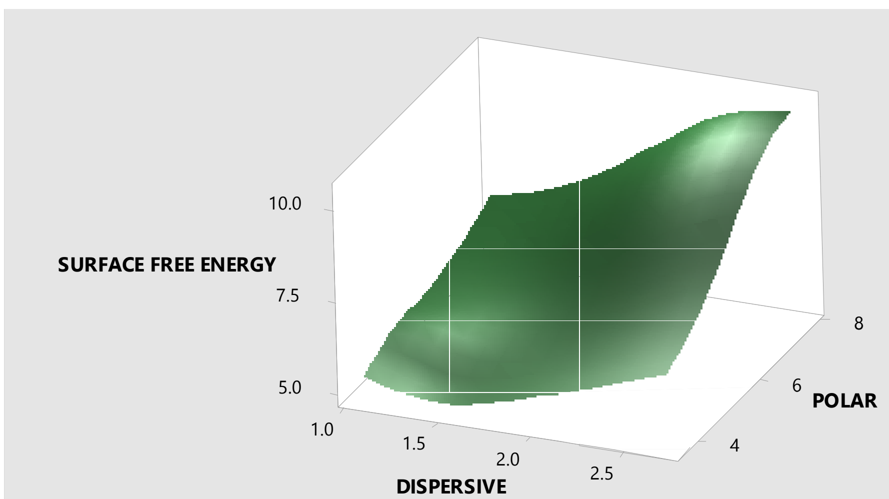


**Fig 8: 3D Surface Plot of Surface Free energy vs Polar and Dispersive Surface Free Energy for Silicon**

Fig 8 shows that there exists a linear relationship between the surface free energy of silicon pressure sensitive adhesive and its dispersive and polar components. The plot shows that a rise in values of the polar component is accompanied by a rise in the corresponding values of the surface free energy, likewise the rise in the value of the dispersive component corresponds to rise the value of the surface free energy. Thus the value of surface free energy for silicon pressure sensitive adhesive is directly influenced by interactions between the polar and dispersive components of the surface free energy.

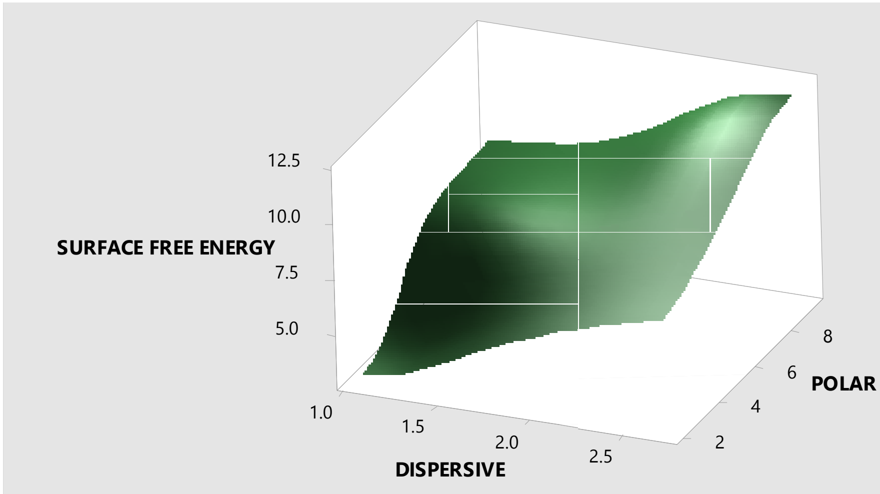


**Fig 9: 3D Surface Plot of Surface Free energy vs Polar and Dispersive Surface Free Energy for Natural Rubber**

Fig 9 shows that the surface free energy has a linear relationship with the polar and dispersive components of surface free energy. The plot shows that for each rise in the values of the dispersive component of the surface free energy, there is a corresponding rise in the polar component, is accompanied by a corresponding rise in the surface free energy.

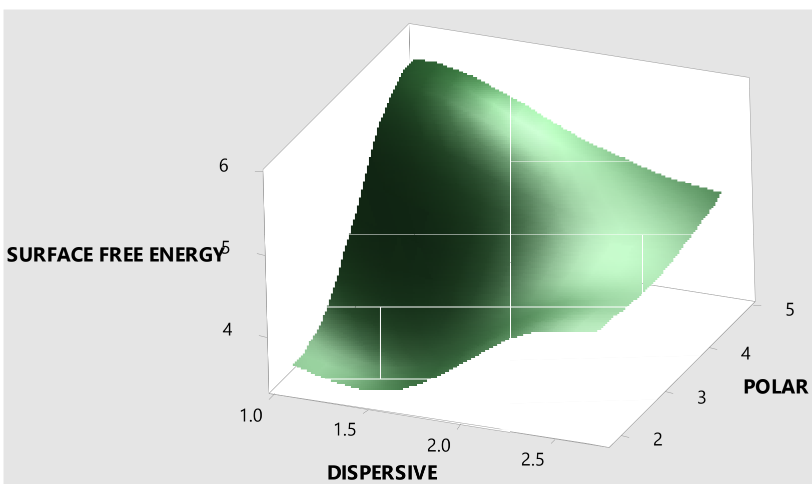


**Fig 10: 3D Surface Plot of Surface Free energy vs Polar and Dispersive Surface Free Energy for Mild Steel**

Fig 10 shows a rise in the surface value of the dispersive component of surface free energy and a corresponding rise in the surface free energy value of the mild steel. The polar component values maintain a uniform (constant) relationship with the corresponding surface free energy value.

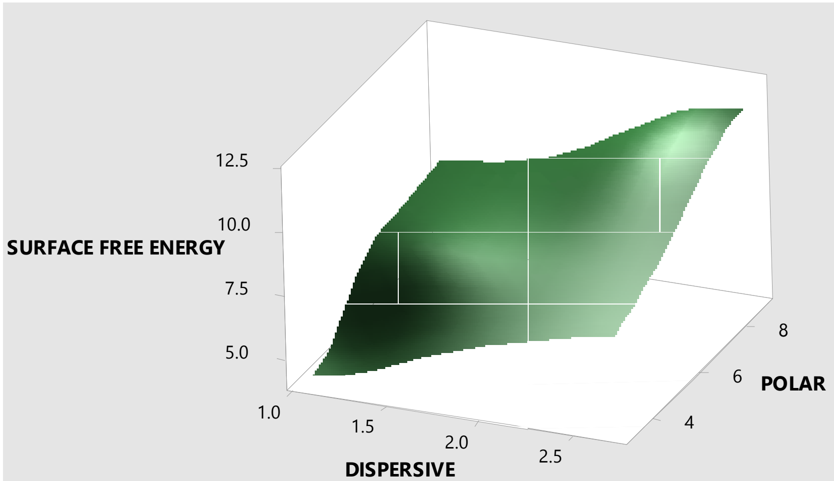


**Fig 11: 3D Surface Plot of Surface Free energy vs Polar and Dispersive Surface Free Energy for Epoxy**

Fig 11 Show that the surface free energy has a linear relationship with the polar and dispersive components of surface free energy. The plot suggests that for noticed rise in value of dispersive component, there is a corresponding rise in the value of surface free energy (Z axis). Likewise for a rise in the values of polar component of the surface free energy, corresponds with the rise in surface free energy. The value of the surface free energy for epoxy pressure sensitive adhesive is directly influenced by the interactions between the polar and dispersive components of surface free energy.

### 4.7 Analysis of substrate surface modifications useful in enhancing interfacial adhesion (between substrate and the adhesive)

The characterization of the various substrate surfaces showed the chemical composition of each substrate surface. This led to a proper identification of each surface as the substrate chemical properties played major role in the interfacial adhesion with pressure sensitive adhesives. It is worth mentioning that the various substrate (walls) studied in this research had earlier been engineered to serve particular primary purposes walls. The hoisting of the emergency sign (Aluminum substrate) bearing materials using designated procedures and abiding by strict regulation comes later. Thus the surface of the substrate need be modified to create a basis for better adhesion and minimizing failure at the interface. The following procedure which involves identification of sources of contamination for the interface substrates and proffering accompanied solution are suggested ways of remediating failure. The entire suggestion is geared toward promoting better adhesion they are as follow:

**Table 4: Substrate Surface and Method of Surface Preparation for Adhesive bonding**

| Substrates | Sources of contamination | Effects on substrate surface | Suggested solution. |
|---|---|---|---|
| Mild Steel | -Oxides<br>- Scales<br>- Paints<br>- Rust<br>- Atmospheric moisture<br>- Organic soil<br>- Cutting fluids | -Rust, oxides, paint, scale inhibit wettability of the surface cause high contact angle formation<br>- Atmospheric moisture encourage formation boundary layer which interne with adhesive wetting of surface<br>- Organic soil encourage befouling | -Degrease<br>- Sand blast<br>- Plasma treatment<br>- Spray cleaning<br>- Abrading<br>- Chemical treatment |
| Aluminum | -Rust protection oil<br>- Grease<br>- Cutting<br>- Finger print<br>- fluid<br>- Mold<br>- scale | Rust, promotion the formation of oxides which inhibit wettability by adhesive and test liquid<br>-grease, cutting fluid, finger print stop intermolecular and chemical bonding<br>-mole encourage befouling which reduce substrate surface energy. | -plasma treatment<br>- spray cleaning<br>- avcaline cleaning<br>- etching with acid<br>- Corona discharge. |

### 4.8 Results and Analysis of Adhesion Prediction

Table 5 shows parameters values derived across bond types with regards to respective pressure sensitive adhesives. Across all adhesives (except cow skin) used for the work, there is relatively noticeable spread ability of each adhesive across interface. This shows good wettability. The work of cohesion exhibited by the cow skin adhesive is greater than its work of adhesion. Thus, low wettability potential and inadequate basis for interfacial bonding as spreading parameters indicate negative. Table 6 shows a logarithm value of the peak force, adhesion ratio and correlation between the two parameters across different bonds type. The adhesion ratio across each bond type shows the potential for tack for respective bond types with regard to each pressure sensitive adhesive. The higher adhesion ratio, gives rise to higher potential of achieving better bonding at interface. The test of a correlation between the values of the logarithm peak force and adhesion ratio showed a range of -1 to +1. Logarithm- linear relationship was used in the work to bring close variables whose numerical values are exceptionally higher than the other variable. The aim is to facilitate comparability. A correlation of +1 between the peak force and the adhesion ration shows that an increase in one variable results in a corresponding increase in the other variable. A correlation of -1 means that both variables move in opposite directions as one variable rises the corresponding variable decreases. A correlation of +1 showed that the adhesion ratio expressed the tack across each bond type. A correlation of -1 shows that there was little or no significant adhesion across that particular bond type.

**Table 5: Results of Force at Peak, Work of Adhesion, Work of Cohesion, Total work of adhesion and Work of Spreading across bond type**

| | Force At Peak (N) | $W_A$ (J/m$^2$) | $W_C$ (J/m$^2$) | $W_{AC}=W_A+W_C$ (J/m$^2$) | $W_S=W_A-W_C$ (J/m$^2$) |
|---|---|---|---|---|---|
| Natural rubber | 1525.1 | 282.91 | 31.44 | 314.35 | 251.47 |
| BOND TYPE | 1239.5 | 199.44 | 31.44 | 230.88 | 168.00 |
| HDF/NR/ALU | 8418.4 | 130.60 | 31.44 | 162.04 | 99.16 |
| CWT/NR/ALU | 1650.9 | 158.19 | 31.44 | 189.63 | 126.75 |
| MS/NR/ALU | | | | | |
| RT/NR/ALU | | | | | |
| EPOXY | | | | | |
| BOND TYPE | | | | | |
| HDF/Epoxy/ALU | 2221.0 | 275.62 | 20.26 | 295.88 | 255.36 |
| CWT/Epoxy/ALU | 419.7 | 195.10 | 20.26 | 215.36 | 174.84 |
| MS/Epoxy/ALU | 7840.9 | 126.19 | 20.26 | 146.45 | 105.93 |
| RT/Epoxy/ALU | 1834.9 | 92.58 | 20.26 | 112.84 | 72.32 |
| Acrylic | | | | | |
| BOND TYPE | | | | | |
| HDF/Acrylic/ALU | 1655.0 | 195.17 | 22.78 | 217.95 | 172.39 |
| CWT/Acrylic/ALU | 1892.3 | 126.59 | 22.78 | 149.37 | 103.81 |
| MS/Acrylic/ALU | 6041.4 | 58.15 | 22.78 | 80.93 | 35.37 |
| RT/Acrylic/ALU | 1581.9 | 56.60 | 22.78 | 79.38 | 33.82 |
| Cow Skin | | | | | |
| BOND TYPE | | | | | |
| HDF/CS/ALU | 167.1 | 46.86 | 108.4 | 155.26 | -61.54 |
| CWT/CS/ALU | 1697.6 | 6.64 | 108.4 | 115.04 | -101.76 |
| MS/CS/ALU | 7120.0 | -28.33 | 108.4 | 79.77 | -136.73 |
| RT/CS/ALU | 1826.0 | -35.60 | 108.4 | 72.50 | -144.00 |
| Silicon | | | | | |
| BOND TYPE | | | | | |
| HDF/SI/ALU | 1746.8 | 163.64 | 14.96 | 178.60 | 148.68 |
| CWT/SI/ALU | 1810.2 | 159.19 | 14.96 | 174.15 | 144.23 |
| MS/SI/ALU | 5783.4 | 96.11 | 14.96 | 111.07 | 81.15 |
| RT/SI/ALU | 1747.1 | 70.02 | 14.96 | 84.98 | 55.06 |

**Table 6: Result of Log of Force at Peak, Adhesion Ratio and Correlation**

| | Logarithm (ln) of Force at peak | Adhesion ratio (A.R) | Correlation |
|---|---|---|---|
| Natural rubber | 7.3298 | 0.8000 | |
| bond type | 7.1225 | 0.7277 | 1 |
| HDF/NR/ALU | 9.0382 | 0.6119 | |
| CWT/NR/ALU | 7.4091 | 0.6684 | |
| MS/NR/ALU | | | |
| RT/NR/ALU | | | |
| Epoxy | | | |
| BOND TYPE | | | |
| HDF/EPOXY/ALU | 7.7057 | 0.8631 | 1 |
| CWT/EPOXY/ALU | 6.0395 | 0.8118 | |
| MS/EPOXY/ALU | 8.9671 | 0.7233 | |
| RT/EPOXY/ALU | 7.5147 | 0.6409 | |
| Acrylic | | | |
| BOND TYPE | | | |
| HDF/ACRYLIC/ALU | 7.4116 | 0.7910 | |
| CWT/ACRYLIC/ALU | 7.5455 | 0.6950 | 1 |
| MS/ACRYLIC/ALU | 8.7064 | 0.4370 | |
| RT/ACRYLIC/ALU | 7.3664 | 0.4261 | |
| Cow Skin | | | |
| Bond type | | | |
| HDF/CS/ALU | 5.1186 | -0.3964 | -1 |
| CWT/CS/ALU | 7.4370 | -0.8846 | |
| MS/CS/ALU | 8.8707 | -1.7141 | |
| RT/CS/ALU | 7.5099 | -1.9862 | |
| Silicon | | | |
| BOND TYPE | | | |
| HDF/SI/ALU | 7.4655 | 0.8325 | 1 |
| CWT/SI/ALU | 7.5012 | 0.8282 | |
| MS/SI/ALU | 8.6627 | 0.7306 | |
| RT/SI/ALU | 7.4657 | 0.6479 | |

## 4.0 Conclusion

The role of interfacial parameters on the adhesion of pressure sensitive adhesives was studied. The inability of the emergency sign (aluminum substrate) to stick on the vertical walls (substrate surfaces) through the use of various pressure sensitive adhesive triggered the necessity for this research.

This is because the inability of the aluminum to stick to the substrate is a function of interfacial phenomenon. The vertical wall substrates are made from mild steel. The adhesives are acrylic, epoxy, silicon, natural rubber material and cow skin (animal hides).

Three test liquids were respectively used to derive the contact angle for each substrate, as well as pressure sensitive adhesive. The contact angles for the substrates range from $55.74^0$ to $163.59^0$while the adhesives range from $78.69^o$ to $181.85^o$.The contact angle enabled the surface free energy for each substrate and each adhesive to be calculated, through Fowkes law. The Kaelble plot was used to derive the values of polar and dispersive component of the surface energy free energy. Owendts law was used to derive interfacial surface energy between substrate and adhesive. The knowledge of the surface free energy from both the adhesives and substrates enable the research analyze the wettability performance of the adhesives on substrates. Young-Drupe equation helped to derive the thermodynamic work of adhesion at various substrate-adhesive interfaces. This was arrived at, through the use of surface free energy value and interfacial energy value in the equation (Young-Drupe). The examination of the adhesive rheology produced evidence of the role of surface polarity (dispersive or polar) in the determination of adhesive strength. This led to the affirmation of the significant impact of electrostatic model of adhesion in determining the thermo dynamic work of adhesion. XRF (x-ray fluorescence) was used to characterize the substrates chemical composition. The adhesive characterization was done to identify its chemical constituents. The process enables the research identify and observe the influence of similarity in chemical properties in promoting interfacial adhesion. Surface roughness was used to measure the surface finish of each substrate. The impact of surface roughness on the thermodynamic work of adhesion was established for each substrate and allied bond pair. The interfacial energy at bond interface was observed to influence the outcome of thermodynamic work of adhesion across the bond. Fracture energy for each bond pair was conducted via mechanical tensile pull-out test. It showed a stress-strain relationship for each adhesive for energy particular bond type. The observation showed various failure modes on each pull-out. Adhesive failure and mixed failure across the bond were characterized by good tack values (nominal stress) and good shear performance. Brittle failure showed good tack but poor shear performance, while cohesive failure showed poor tack and poor shear performance. Bond characterized by adhesive, mixed and brittle failure showed slight to non-homogenous debond. Cohesive failure showed slight to noticeable homogenous debond. Analytical tools such as Minitab-3D surface plot, SPSS and Microsoft excel were used in the research. The Excel and SPSS observed an inverse relationship between the thermodynamic work of adhesion and fracture energy. Excel scatter plots showed a regression analysis of 0.5 to 1 for the adhesive and 0.4944 to 1 for substrate. SPSS Pearson correlation showed that 50% of variation in the value of polar component of surface free energy can be attributed to the dispersive component of the surface free energy. The Minitab 3D surface plot showed the relationship between the value of the dispersive and polar component in determining the value of the surface free energy. For substrates the relationship ranges from linear relation, inverse relation to partial relation. For the adhesives the relationship ranges from linear to partial. Surface modification procedures were suggested to improve adhesion performance for substrate and adhesive at interface. This research concluded that interfacial parameters indeed influence the adhesion of the emergency sign on the substrate (vertical walls) in the presence of pressure sensitive adhesives. Hence establishing the fact that when substrate and pressure sensitive adhesives of opposite polarities are bonded at interface, stronger work of adhesion is achieved (This extends the hypothesis that close ratio matching of polarity components between adhesive and substrates predicts stronger adhesion).

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