

Use of Surface Energy to evaluate Adhesion of Bituminous Crack Sealants to Aggregates

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Abstract: Problem statement: The performance of bituminous sealants in the field is partly controlled by properties and strength of the sealant-aggregate interface. The thermodynamic work of adhesion represents energy required for reversible separation of the two materials at the interface. The study of fracture includes the energy required for both reversible and irreversible processes during separation. Excess of work of fracture over work of adhesion represents the energy consumed by irreversible processes in the specimen during loading and fracture. In addition, adhesion strength is related to the properties of constitutive components. **Approach:** The objective of this study was to measure or predict adhesion of hot-poured bituminous-based sealant to aggregates of different chemical composition. In order to accomplish, the study of fracture and the thermodynamic work of adhesion were estimated for 14 hot-poured bituminous-based sealants with two types of aggregate: Limestone and quartzite. The work of adhesion for each sealant-aggregate system was calculated from the contact angle measurements of system components at corresponding sealant installation temperature. **Results:** In general, limestone showed better adhesion to hot-poured crack sealant than quartzite. Interfacial parameters such as contact angles and surface tensions were successfully used to differentiate between sealants. **Conclusion/Recommendation:** The findings were in agreement with preliminary testing results of 14 sealants using a newly developed fixture in the direct-tension testing machine. Provided the sealant has an appropriate viscosity to fill the crack, as the sealant surface tension decreases, its adhesion strength increases.

Key words: Adhesion strength, contact angle, surface tension, crack sealant, hot-poured bituminous, solid surface, Young-Dupré equation, Strategic Highway Research Program (SHRP), higher temperature

INTRODUCTION

Adhesion is a fundamental property of a sealant/aggregate's interface. It is a phenomenon that depends directly on interatomic and molecular forces between the adherent and the substrate (Masson *et al.*, 2002; Al-Mutairi *et al.*, 2009; Kumar and Vidivelli, 2010; Atar and Colakoglu, 2009). The adhesion strength is affected by wetting and interlocking. Wetting is controlled by the attraction forces between the two surfaces - solid and liquid and it shows how the substrate interacts with liquid (Fini and Abu-Lebdeh, 2011). For example, in case of sealant-aggregate (or HMA) interaction, in which the aggregate is considered a solid and the sealant a liquid, adhesion strength is strongly affected by the composition and surface chemistry of the two. Aggregate characteristics show a stronger effect compared to

adhesive. A convenient method to measure the wetting of a solid surface is to determine the contact angle of a liquid, which is formed when a drop of liquid is placed on a perfectly smooth and rigid solid (Fini and Abu-Lebdeh, 2011; Guangyang *et al.*, 2010). Amin and co-workers (Amin *et al.*, 2010) investigated the wettability of several substrate surfaces and determined the contact angles on each substrate by observing the spreading area of the water droplets that referred to the surface tension of the droplets towards the substrate surface.

Because adhesion strength is controlled mainly by wetting and interlocking, it can be evaluated through contact angle measurement by using the Young-Dupré equation. However, in order to measure the adhesion strength of the interface quantitatively, a mechanical test is needed. Nevertheless, conventional tests measure

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bond strength, which can be just an estimation of the adhesion strength. Bond strength actually depends on both interfacial and bulk characteristics of the assembly. Masson and Zanzatto independently measured the bond strength of sealants in Portland cement concrete. They used a direct tensile test and brought the sealant-PCC assemblies to tensile failure. They measured bond strength of 200-500J m⁻² at -37°C and 500-1000J m⁻² at -30°C, respectively. Although results showed higher bond strength at the higher temperature, it doesn't necessarily indicate higher adhesion strength at higher temperature. It can be argued that the sealants extended more at higher temperatures and dissipated more energy, which is more of a bulk effect (Masson and Lacasse, 2000). In Masson and Lacasse, both portable and full scale adhesion tests were used to assess the level of sealant adhesion to heat-treated substrates. They used the direct tension method to measure the bond strength of the sealant-HMA briquette assemblies. They calculated the area under the stress-strain curve as the bond strength. The full scale test was used to simulate field conditions and to validate the portable testing results. Test results were indicative of sealant performance in cold temperatures. Further, several studies on the binder-aggregate adhesion have been conducted. For instance, tensile tests were performed on asphalt-concrete (HMA or PCC) blocks as part of the Strategic Highway Research Program (SHRP). These qualitative tests provided an indication as to the material's ability to extend without experiencing cohesive and/ or adhesive failure. They showed that sealants bonded to HMA blocks normally incur greater stresses than sealants bonded to PCC. Youtcheff and Aurilio (1997) tried to measure the bonding strength of asphalt binder applied to a flat surface, using a Pneumatic adhesion tester, while Kanitpong and Bahia (2005) proposed a modified pull-off test, which applied tensile force on the binder/ aggregate assembly to measure adhesion characteristics of selected asphalt binder to various mineral surfaces.

In general, two key requirements must be met when using a mechanical test to study the adhesion between two dissimilar materials. The first is to use a mechanical test that yields a system parameter which is independent of the material's geometry. The second is to achieve failure at the interface between the two materials so that conclusions can be drawn about adhesion rather than cohesion. For example: In the direct tension test, a system parameter is not readily extractable from test results because the results depend

on specimen geometry and the elastic properties of the adhering material. Hence, such a test can be used only for quality control applications or comparison of surface treatments, not for determining a fundamental property of the material (Masson *et al.*, 2002). In the Strategic Highway Research Program (SHRP) (1994) reported that the application of fracture mechanics techniques to measure and predict binder-aggregate bond strength could be promising. A blister test, which is used in polymer research, was evaluated. The research concluded that the test did not reflect field conditions of adhesion failure and could not differentiate between various aggregates (Fini and Abu-Lebdeh, 2011). Masson and Lacasse (2000) tried to measure adhesion using electrical resistance. They measured the resistance-to-mass transfer along the sealant-HMA interface and the rate of the resistance changes; but for this method to yield reliable results, the resistance to wetting relation must also be established. If the surface energy of an HMA, or aggregate and that of a sealant were known, the work of adhesion at the interface could be calculated. Although to predict bond strength, the measurement of a number of other parameters, such as roughness and absorption, is required, the study of adhesion as a fundamental characteristic of the interface can be used to estimate the strength of the bond. In this study, to determine the sealant-aggregate adhesion, the study of adhesion of 14 hot-poured sealants onto two aggregate types was determined. The study of adhesion was calculated utilizing the physical chemistry theory developed by van Oss *et al.* (1988).

MATERIALS AND METHODS

Adhesion at the interface: Adhesion strength can be evaluated through contact angle (Fig. 1) which is defined geometrically as the angle formed by a liquid at the three phase boundary where a liquid, gas and solid intersect. Contact angle formed between the droplet of the liquid and the solid surface is well defined quantity which can be used to calculate the study of adhesion through the Young-Dupré equation. As can be seen in Fig. 1, low values of θ indicate that the liquid spreads and wets the surface easily, while higher values show poor wetting. After a liquid wets the surface of the solid and fills the tiny pores, it solidifies and develops what is called interlocking. This interlocking is strongly affected by the liquid's viscosity at installation. Low viscosity sealant can penetrate and follow the surface irregularities better than high viscosity sealant and, hence, results in a better interlocking (Penn and Defex, 2002).

Two very common, yet different, approaches exist to measure contact angles between different liquids and non-porous solids: goniometry and densitometry.

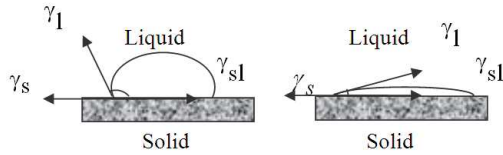


Fig. 1: Contact angle of a liquid onto a solid

The goniometry method is basically the analysis of a drop of test liquid on a solid substrate. The components of a goniometer are a light source, sample stage, lens and image capture. Contact angle can be measured directly by examining the angle formed between the solid and the tangent to the drop surface (Fig. 1). To use the Young-Dupré equation, measurement of the surface tension of the liquid, γ_l , surface free-energy of the solid, γ_s and the solid-liquid interfacial free-energy, γ_{sl} , are required. Knowing γ_l and γ_s (discussed later in this study), the third component can be calculated through the Young's equation:

$$\gamma_{sl} = \gamma_s - \gamma_l \cos \theta \tag{1}$$

Where:

- γ_{sl} = The solid-liquid interfacial free-energy (mJ m^{-2})
- γ_s = The surface free-energy of the substrate (mJ m^{-2})
- γ_l = The surface tension of the liquid (mJ m^{-2})
- θ = The contact angle ($^\circ$)

Work of Adhesion is defined as the study required to separate the liquid and solid phases. Basically, when two dissimilar materials form an interface by being in intimate contact, a tensile force can be applied to split the materials into dissimilar parts. For a completely brittle interface of unit cross-sectional area, the energy expended (or work of adhesion) can be obtained using the Young-Dupré equation:

$$W_a = \gamma_s + \gamma_l - \gamma_{sl} \tag{2}$$

Substituting Eq. 1 in 2:

$$W_a = \gamma_l \cos \theta + \gamma_l = \gamma_l (1 + \cos \theta) \tag{3}$$

In order to use the above equation, one needs to measure the surface tension of the liquid, γ_l . Knowing that surface tension is comprised of a polar and non-polar component (Fini *et al.*, 2008), the surface tension of a single phase (liquid in this case) may be presented as follows:

$$\gamma_l = \gamma^{LW} + \gamma^{AB} \tag{4}$$

Where:

- γ_l = The liquid surface tension (mJ m^{-2}); and
- γ^{LW} = Denotes the Lifshitz-van der Waals and
- γ^{AB} = The acid-base component

Van Oss and his co-workers, incorporating the surface energy of both solid and liquid phases, described in polar and non polar components, presented the full version of the Young-Dupré equation as follows:

$$W_a = (1 + \cos \theta) \gamma_l = 2[(\gamma_s^{LW} \gamma_l^{LW})^{\frac{1}{2}} + (\gamma_s^+ \gamma_l^-)^{\frac{1}{2}} + (\gamma_s^- \gamma_l^+)^{\frac{1}{2}}] \tag{5}$$

$$\gamma_s^{\text{total}} = \gamma_s^{LW} + 2[(\gamma_s^+)(\gamma_s^-)]^{\frac{1}{2}} \tag{6}$$

Where:

- γ_s^{LW} = The Lifshitz van der Waals or non-polar component;
- γ_s^+ = The acid or electron accepting component; and
- γ_s^- = The base or donating electron donating component.

While γ_s^{LW} , γ_s^+ and γ_s^- are the components of the surface free energy of the solid, γ_l^{LW} , γ_l^+ and γ_l^- are those of the liquid and θ is the contact angle.

Using the aforementioned equation, one can predict the study of adhesion between two materials if their surface energy components are known. The condition of components at the time of wetting is also very important. For example: When asphalt binder is applied to hot aggregate, it binds stronger than when applied to cold aggregate. When aggregate with absorbed water film is heated, water vapor escapes and it loses its outermost absorbed water molecules. Hence, its interfacial tension-to-asphalt-binder decreases and results in developing a stronger bond between the two. The stripping in HMA is caused by the presence of free-energy.

Experimental program: The sealant wetting is largely controlled by its viscosity. While a low viscosity sealant greatly facilitates wetting, a high viscosity sealant facilitates adhesion once wetting has been secured. A sealant with low viscosity is much more readily removed from an aggregate surface than a high viscosity sealant; this is particularly true if the surface is smooth. Another important factor that influences wetting is the surface tension of the sealant. In this study, sealant viscosity and contact angle, sealant and aggregate surface tension and surface energy components were calculated to determine the sealant-aggregate adhesion.

Table 1: Viscosity measurements using brookfield viscometer at pouring temperature and stirring speed of 60 rpm

Product	Temperature (°C)	Viscosity (Pa. s)
BB	193	1.75
DD	193	4.21
MM	170	1.66
NN	185	6.10
PP	193	3.00
V V	149	0.99
WW	188	2.58
AD	188	0.35
AE	188	1.64
UU	193	2.53
EE	193	1.79
QQ	193	5.11
YY	177	6.96
ZZ	193	4.16
AB	177	6.01

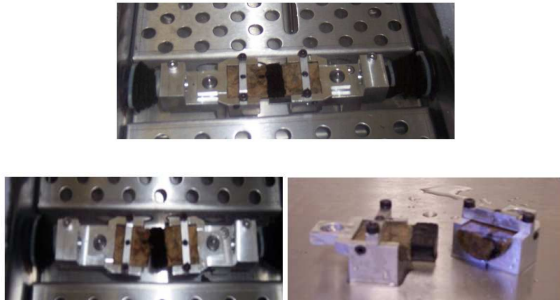


Fig. 2: Illustration of the newly-developed adhesion test fixture

The viscosity of 14 hot-poured crack sealants was determined at the manufacturers' recommended installation temperatures. SuperPave's recommended viscosity test was modified to accommodate the testing of crack sealants, which have high polymer contents. The modified procedure has been reported elsewhere (Al-Qadi *et al.*, 2007). The viscosity results are presented in Table 1. Further, the sessile drop method was used to determine the surface tension of the hot-poured crack sealants. A thin layer of each sealant, heated to installation temperature, was prepared and allowed to cool to room temperature. Then a five-micrometer pipit was used to manually apply a droplet of three probe liquids - water, form amide and glycerol - on the sealant and the images of the drops were captured by microscope within 15s of application. The wetting values of the surface were reported either as contact angle or by the cosine of the contact angle and plugged into Eq. 5.

Fracture energy measurement: A preliminary testing fixture was developed to assess sealant-aggregate adhesion utilizing the Direct Tension Tester (DTT).

The briquette assembly consists of two half-cylinder aggregates of 25mm diameter and 12mm length. The assembly has a half cylinder mold, open at the upper part. Prior to pouring the sealant, the assembly is heated to facilitate sealant flow and to ensure a uniform bonding area. The aspect ratio (width to depth) of the sealant is maintained 1 (Fig. 2). A pre-deboned area was applied in form of a notch at the upper edge of the interface and placed at one side of the assembly. After 1h of curing, the specimen was trimmed and kept in the DTT cooling bath for 30min before testing. Because of the pre-deboning area, resembling an initial crack, no energy is dissipated in the crack initiation. In addition, the failure path is defined. This approach resulted in consistent results compared to those of unnotched specimens (Fig. 2).

RESULTS

The energy components of the probe liquids (Table 2) results in three equations and three unknowns, which represent the energy components of the sealant: $\gamma_{Sealant}^{LW}$, $\gamma_{Sealant}^+$, $\gamma_{Sealant}^-$. Hence, using Eq. 6, surface tension of the sealant, $\gamma_{Sealant}^{total}$, can be calculated.

The surface tension values for the 14 tested sealants are presented in Table 3. However, to calculate the study of adhesion, the contact angle between the sealant and the aggregate is needed. A small pin was used to manually apply a droplet of sealant heated to installation temperature to the aggregate. The aggregate surface was cleaned with acetone and distilled water and then dried in oven. The contact angle between each individual pair of sealant-aggregate was measured by the same method as described before and presented in Table 4. Using Eq. 3, work of adhesion, W_a , was calculated for each pair. The 14 sealants were tested with two aggregate types, limestone and quartzite. The testing results are presented in Table 5.

Fracture energy measurement: Using the fracture energy testing approach, the aforementioned 14 sealants were tested with quartzite, (Table 6). Tests were conducted at the lowest possible application temperature (-4 to -34°C), depending on sealant type and at a cross-head speed of 0.5 mm sec⁻¹. Under these testing conditions, the sealant behaves as a rigid element and the area under the stress-strain curve can be regarded as an estimation of the energy to rupture or fracture energy. Fracture energy is related to the study of adhesion. It has been experimentally observed that fracture energy increased exponentially with the study of adhesion.

Table 2: Surface energy characteristics of probe liquids at 20°C, J m⁻²

Probe liquid	γ_l	γ_l^{LW}	γ_l^+	γ_l^-
Water	72.8	21.8	25.5	25.5
Glycerol	64	34	3.92	57.4
Formamide	58	39	2.28	39.6

Table 3: Surface tension and viscosity of sealants and water

Sealant	γ_l^{LW}	γ_l^+	γ_l^-	γ_{total}	Viscosity (Pa.s)
EE	0.10	4.88	18.72	19.22	1.79
ZZ	10.03	5.17	0.39	12.89	6.96
YY	2.89	10.03	2.40	12.70	1.75
UU	2.39	6.32	4.75	13.35	2.53
VV	32.45	2.18	0.84	35.15	0.99
AE	43.99	5.21	4.36	53.53	1.64
DD	18.28	4.38	0.29	20.52	4.21
AD	34.49	5.68	1.84	40.96	0.35
WW	9.03	18.80	1.20	18.54	2.58
MM	45.15	12.01	4.93	60.54	1.66
PP	13.98	4.40	0.60	17.22	3.00
QQ	0.01	5.39	11.60	15.83	5.11
NN	6.63	19.99	0.37	12.06	6.10
BB	9.97	40.01	0.94	22.25	1.75
Water	9.97	0.94	40.01	72.8	Very low

Table 4: Contact angle between different liquids and substrates

Liquid/Substrate	Limestone	Quartzite	Sandstone
EE	136	137	135
ZZ	106	139	86
YY	127	173	63
UU	84	105	94
VV	136	145	91
AE	143	132	134
DD	126	153	141
AD	82	150	75
WW	71	76	81
MM	90	133	118
PP	112	128	102
QQ	141	167	176
NN	108	133	120
BB	70	61	63
Water	30	65	-
Glycerol	87	74	41
Formamide	7	69	-

Table 5: Interfacial surface tension and work of adhesion of different aggregate/sealant combinations

Liquid/substrate	Limestone		Quartzite	
	γ_{sl}	W_a	γ_{sl}	W_a
EE	27.30	5.39	71.94	5.16
ZZ	17.10	9.26	67.66	3.11
YY	21.06	5.12	70.49	0.09
UU	12.00	14.82	61.41	9.82
VV	38.90	9.72	86.68	6.36
AE	56.03	10.97	93.70	17.71
DD	25.63	8.36	76.22	2.18
AD	7.77	46.66	93.36	5.49
WW	7.33	24.68	53.40	23.03
MM	13.12	60.89	98.91	19.51
PP	19.92	10.77	68.56	6.54
QQ	25.78	3.53	73.29	0.43
NN	17.13	8.40	66.16	3.79
BB	5.98	29.74	47.10	33.03
Water	-49.36	135.63	26.73	103.95
Glycerol	9.75	67.72	39.88	82.00
Formamide	-44.05	115.53	37.10	78.79

Table 6: fracture energy and surface tension for fourteen sealants with quartzite

	Surface Tension (mJ m ⁻²)	Viscosity (Pa.s)	T of test (°C)	Fracture energy (J m ⁻²)
EE	19.22	1.79	-10	3.13
MM	60.54	1.66	-34	3.31
ZZ	12.89	6.96	-10	3.33
VV	35.15	0.99	-34	4.85
DD	20.52	4.21	-34	4.88
AD	40.96	0.35	-34	7.29
AE	53.53	1.64	-34	8.01
QQ	15.83	5.11	-4	9.15
WW	18.54	2.58	-34	12.33
PP	17.22	3.00	-34	22.44
YY	12.70	1.75	-10	25.01
NN	12.06	6.10	-34	25.59
UU	13.35	2.53	-10	45.44
BB	22.25	1.75	-34	228.99

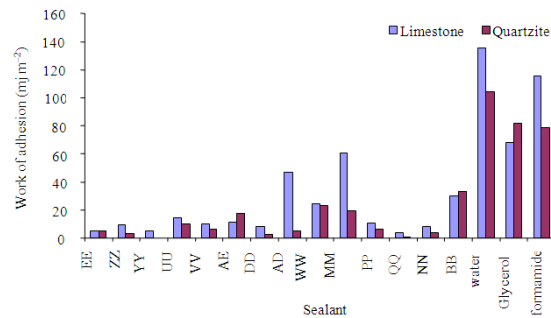


Fig. 3: Work of adhesion of different aggregate/sealant assemblies

Interfacial parameters: Interfacial parameters such as contact angles and surface tensions were successfully used to differentiate between sealants. The hot-poured bituminous-based sealant adhesion to aggregate appears to be a function of viscosity and surface tension of the sealant. The lower the surface tension and viscosity, the greater the adhesion strength. In general, limestone showed better adhesion to hot-poured crack sealant (Fig. 3). In spite of the high variability in the sealant composition and sealant polymer content, surface energy measurements appear to adequately differentiate between sealants.

Using a newly developed fixture in the direct tension-testing machine, limited adhesion testing of 14 sealants supports the aforementioned findings. Figure 4 shows the estimated fracture energy vs. surface tension of those sealants whose viscosity value is lower than 3Pa.s and were tested at -34°C. Statistical analysis shows a nonlinear relationship between surface tension and fracture energy (Fig. 4). It appears that lower surface tension leads to higher fracture energy; hence, increases wet ability and adhesion strength.

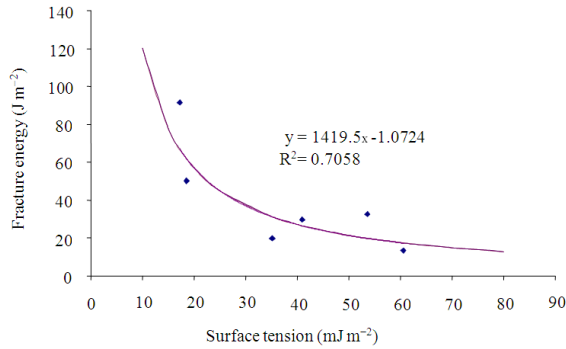


Fig. 4: Fracture energy vs. surface tension for sealants with viscosity below 3Pa.s at -34°C

A nonlinear regression for sealants having a viscosity lower than 3Pa.s was performed. The R^2 was found to be 0.705; this value represents the portion of variation in fracture energy that can be explained by the surface tension of the crack sealant.

DISCUSSION

In this study, sealant viscosity and contact angle, sealant and aggregate surface tension and surface energy components were calculated to determine the sealant-aggregate adhesion. The concept of fracture in materials was divided into two general classifications: (i) the separation of a material from itself (cohesive fractures); and (ii) the separation of a material from a dissimilar material at the interface surface between the two materials (adhesive fracture). To evaluate the adhesion strength between bituminous (hot-poured) sealants and Hot-Mix Asphalts (HMAs) or aggregates, several aspects of the sealant-aggregate bond must be considered. These include molecular characteristics at the sealant-substrate interface, microscopic surface topography and crystallization, macroscopic joint strength and fracture energy. Among the important parameters that control the adhesive strength at the interface are the surface tensions of the materials coming together and the associated free energy of adhesion (Yusilawati *et al.*, 2010), which can be more simply defined as the study required to take apart an interface. Thus, fracture energy is related to the study of adhesion. It has been experimentally observed that fracture energy increased exponentially with the study of adhesion.

Molecules inside a liquid/ solid are affected by equal attraction forces, whereas the molecules at the surface do not have any neighbors on the side toward the air. Therefore, they have greater attraction forces toward the liquid/solid than toward the air. This leads to existence of unbalanced energy, or excess free energy, which is a fundamental property of any liquid or solid.

In the case of liquid it is called surface tension (Bari *et al.*, 2010; Kammuang-Lue *et al.*, 2010); while for solid, surface free energy is a more common name. Surface tension is considered an important parameter to elucidate sealant-HMA adhesion. The surface tension of a liquid is related to the attraction forces between the molecules that make up the liquid. When liquid is poured on a substrate surface, it spreads-out. This indicates that the surface tension holding the liquid molecules together has been overcome. Several methods are available to measure a liquid's surface tension. Methods to measure a solid's surface tension, however, are less common. The surface tension is a function of the substrate surface energy and the substrate surface energy is a function of the forces holding its molecules together. Also, the lower the surface tension and viscosity, the greater the adhesion strength. Statistical analysis of the present study shows a nonlinear relationship between surface tension and fracture energy (Fig. 4). It appears that lower surface tension leads to higher fracture energy; hence, increases wet ability and adhesion strength.

CONCLUSION

The study of fracture and the thermodynamic work of adhesion were determined for 14 hot-poured bituminous-based sealants with two types of aggregate: limestone and quartzite. The study of adhesion was determined from the contact angle measurements of the sealant at installation temperature. Fracture energy was estimated from the direct tension test. The following observations and conclusions can be drawn from these results:

- Interfacial parameters such as contact angles and surface tensions were successfully used to differentiate between sealants
- The adhesion strength is affected by wetting and interlocking
- The lower surface tension leads to higher fracture energy; hence, increases wet ability and adhesion strength. Thus, the lower the surface tension and viscosity, the greater the adhesion strength
- It was observed that fracture energy increased exponentially with the study of adhesion
- Limestone showed better adhesion to hot-poured crack sealant than quartzite

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