Factors Affecting Adherometer Adhesion

The Work of Removal of Organic Coating

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AN EVALUATION of adhesion of an organic coating requires consideration of three regions in which failure in adhesion may occur: the substrate, the interphase, and the coating.

Surface preparation of metallic substrates for industrial finishes has been improved to the extent that the failure in adhesion of an organic coating to a metallic surface in the testing process should take place not in the substrate but in the interphase or region of transition between the metallic substrate and the body of the organic coating. The use of the term "hesion" (*5*) avoids the misleading inference when either of the terms, cohesion or adhesion, is used (*1*).

A unique feature of the ivory knife Interchemical Adherometer as developed by Green and Lamattina (*8*) and Rolle and Dietrich (*11*) was the method for correcting for tear resistance of the coating, friction on the knife, and resistance to plastic flow or pushing action of the knife aganist the bulk of the film by extrapolation of the corrected stripping force to infinitesimal thickness.

In the use of the Adherometer for investigation of the hesion of lacquers to aluminum, the discovery of negative intercepts or "negative adhesion" in the extrapolation of the stripping force curves (*3, 4*) resulted in the use of "mil-hesion," the corrected stripping force at a coating thickness of 1 mil, and the slope, or increase in stripping force per mil increase in thickness. Because of the linear relation found between mil-hesion, the slope and coating thickness for a series of ethylcellulose coating (*3*), and because of the difficulty in determining the components of the stripping force actually responsible for removing the coating from the surface, Adherometer measurements are reported as "work of hesion," the work required to remove a unit volume of the coating from the surface. The work per unit volume is calculated by multiplying the mil-hesion, or the slope of the stripping force curve in units of 10^5 dynes per mm. per mil, by 4.02 to obtain kilogram-meters per cubic centimeter. Work of hesion provides a common basis for comparison of hesion measurements made by different methods.

EXPERIMENTAL

Effect of Rate of Stripping. Wolf (*12*) used an instrument resembling the ivory knife Adherometer to investigate a plasticized nitrocellulose resin lacquer. He reported that the slope of the stripping force curve plotted aganist coating thickness was only sligthly influenced by differences in stripping speeds from 0.024 to 47 inches per minute. However, the extrapolated value of the stripping force, which was reported as adhesion, increased almost as an exponential function of the speed, in disagreement with Green and Lamattina (*8*).

The effect of rate of stripping was investigated in this laboratory with the Interchemical Adherometer modified with a strain gage circuit to record stripping force on a chart recorder, a hardened tool steel knife lapped in place, and a variable speed torque converter to replace the fixed stripping speed of 12 inches per minute with speeds adjustable from 0.1 to 20 inches per minute (*1*).

In general agreement with the results of Wolf, Table I shows that, for a 10-fold increase in the rate of stripping of an 0.82-mil thick castor oil–ureaformaldehyde (60 to 40) baked coating on steel, the slope and the corresponding work of hesion are constant within experimental error, but the limiting stripping force intercept, which has been interpreted as adhesion, decreases by 70%. Coconut oil modified-alkyd, soybean oil modified-alkyd and vinyl baked coatings gave results similar to the castor oil–ureaformaldehyde coating.

Table I. Independence of Work of Hesion and Slope of Stripping Force–Thickness Curve on Rate of Stripping

Rate of Stripping, In. / Min.	Intercept, 10^5 Dynes/Mm.	Slope, 10^5 Dynes /Mm./Mil	Work of Hesion, Kg.-M. /Cu. Cm.
2.0	0.80	1.45	5.8
0.5	0.50	1.35	5.4
0.2	0.25	1.33	5.4

INFLUENCE OF ENVIRONMENTAL CONDITIONS ON HESION

Effect of Drying Conditions. A nitrocellulose lacquer was prepared with 10% tritotyl phosphate plasticizer, in an 85% butyl acetate–15% ethyl alcohol solvent mixture. Aluminum panels were dip coated, air dried for 72 hours, and then dried at 65° C. for 60 hours. The coatings were tested with the Adherometer at different lengths of time after removal from the accelerated drying chamber. The decreased work of hesion with ageing computed from the slope of the stripping force–thickness curves in shown in Figure 1,*A*.

Effect of Temperature. Aluminum panels coated with the same nitrocellulose lacquer were dried for one month under ambient conditions. The panels were then divided into

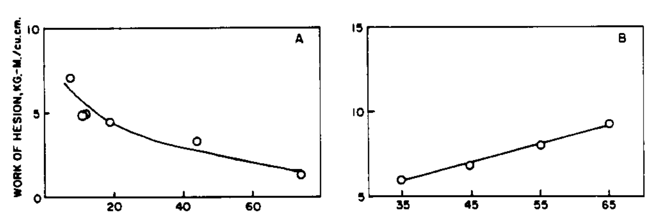

Figure 1. Effect of environmental conditions on hesion using nitrocellulose
A. Drying time B. Temperature

groups to be conditioned at a prescribed temperature for 96 hours. The stripping force was determined immediately at the temperature at which the coating was conditioned. The Adherometer testing assembly was maintained at these temperatures by the use of infrared lamps. The results are shown in Figure 1,*B*. Consistent results were also obtained with coated panels dried for 28 days at 50% R. H. and 30° C. and then divided into three groups to be conditioned at 50°, 60°, or 70° C. for 48 hours (*1*).

In both experiments, an increase in work of hesion was found for an increase in temperature. Lasoski and Kraus (*9*) found the opposite effect for poly(vinyl acetate). Eley (*7*) reported, from a personal communication with De Bruyne, that the tensile failing load of redux joints decreased with an increase in temperature, except for an initial rise in stripping load with an increase in temperature, for which he suggested this explanation: As the temperature was raised the increased elasticity in the adhesive allowed the strain energy to spread over a larger volume of adhesive. McLaren and Seiler (*10*) found the stripping load to increase to a maximum as the temperature was increased for stripping tests of cellulose films held together with vinyl polymers.

EFFECT OF COATING COMPOSITION ON HESION

Effect of Plasticizer and Resin content. The effect of plasticizer content of a nitrocellulose and of an ethylcellulose lacquer was reinvestigated on the basis of work of hesion. Earlier results, interpreted with the limiting stripping force method, had indicated an optimim plasticizer content (*6*).

A series of nitrocellulose lacquers with 80% *n*-butyl acetate was prepared with a range of dibutyl phthalate plasticizer content of the coating from 0 to 50%. Coatings on 24ST aluminum were tested for hesion after drying in air at room temperature for 2 months. The results are shown in Figure 2,*A*. The results for a series of ethylcellulose coatings on aluminum panels prepared in the same way, are shown in Figure 2,*B*.

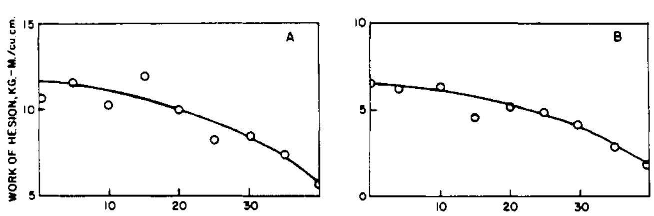

Figure 2. Effect of percentage of plasticizer on hesion to aluminum

A. Nitrocellulose *B*. Ethylcellulose

A series of lacquers containing 15% nonvolatile matter, consisting of ethylcellulose, dioctyl phthalate, and dammar resin (dewaxed) in a solvent mixture of 70% toluene and 30% ethyl alcohol by weight, was prepared in a statistical design. Aluminum panels were wedge-coated and dried for 24 hours at room temperature, then at 65° C. in a circulating air drying chamber for 48 hours, and stored at 25° C. and 50% relative humidity until tested. Work of hesion was calculated from the slope of the stripping force–thickness curves derived from linear regression analysis. Figure 3,*A* and *B*, show that the effect of percentage of plasticizer on hesion for 0 and 20% dammar content is to decrease the work of hesion as the percentage of plasticizer is increased. The effect of the dammar resin is to increase the hesion of the coatings (*1*). Wolf (*12*) also found that for nitrocellulose lacquers plasticized with dibutyl phthalate, the slope of the stripping force curves decreased with an increase in plasticizer content.

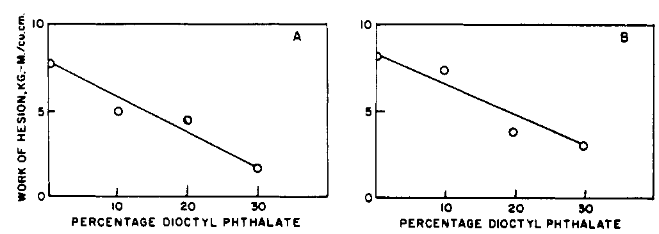

Figure 3. Effect of percentage plasticizer on hesion of pigmented lacquers to aluminum

A. 0% dammar *B*. 20% dammar

Effect of Pigment Content. A series of lacquers was prepared to contain 15% by weight nonvolatile matter, consisting of unplasticized ethylcellulose, talc, and calcium carbonate, in a solvent mixture of 70% toluene and 30% butanol. Aluminum panels were coated and treated in the same way as in the case of the dammar coating (*1*).

Figures 4,*A* and *B*, show that the presence of calcium carbonate decreases the hesion in contrast to an increase found with talc. When used in combination, the effect on hesion was essentially an average of the separate effects of the two pigments.

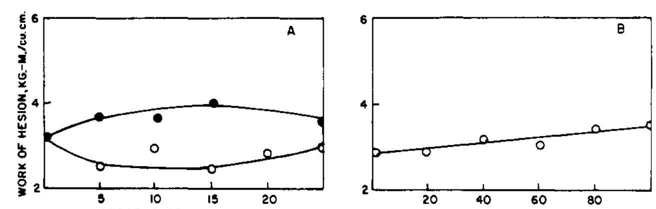

Figure 4. Effect of pigments on hesion of ethylcellulose to aluminum

0 Calcium carbonate

0 Talc

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