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Measuring Adhesion and Cohesion of Rubberized Asphalts

by O. Dutt, H.E. Ashton and H.O. Laaly

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RÉSUMÉ

L'adhérence et la cohésion sont des caractéristiques importantes des bitumes caoutchoutés utilisés pour les revêtements de toiture et l'imperméabilisation. L'essai de dureté-ténacité classique effectué à l'aide d'une tête de traction hémisphérique ou filetée fournit des résultats qui représentent une combinaison des forces d'adhésion et de cohésion. Dans nombre de cas, c'est l'adhérence qui fait défaut, affectant du même coup les valeurs des charges de crête. Une nouvelle tête rainurée et à section décroissante conçue pour améliorer l'adhérence a été testée sur quatre échantillons commerciaux de bitumes caoutchoutés. On a comparé les résultats à ceux obtenus avec les autres têtes. On a effectué la plupart des essais à plusieurs vitesses de chargement afin d'en déterminer l'effet sur les valeurs de dureté et de ténacité. Lors d'une deuxième série d'essais, on a supprimé l'effet de l'adhérence afin de n'évaluer que la cohésion. Enfin, des essais de ductilité et des essais de mesure de l'adhérence par traction ont été réalisés sur chaque échantillon. Les résultats obtenus indiquent qu'il faudrait réévaluer les actuelles méthodes d'essai et exigences numériques visant les propriétés physiques connexes, dans les normes concernant les bitumes caoutchoutés appliqués à chaud.

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Measuring Adhesion and Cohesion of Rubberized Asphalts

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ABSTRACT: Adhesion and cohesion are important aspects of performance of rubberized asphalts used for roofing and waterproofing. The conventional toughness-tenacity test performed with either the hemispherical or screw-type pull-head gives results that represent a combination of adhesive and cohesive forces. In many cases adhesion is lost, thus affecting peak load values. A new, tapering, grooved head designed to improve the adhesion aspect has been tested with four commercial samples of rubberized asphalt. The results are compared with those for other heads. Most tests were conducted at several speeds of loading to study the effect on toughness and tenacity values. In a second series of tests, the effect of adhesion was eliminated and only cohesion determined. Finally, straight pull-off adhesion tests and ductility tests were conducted on all samples. The results suggest that the present accepted test methods and numerical requirements for the related physical properties in standards for hot-applied rubberized asphalts should be reassessed.

KEY WORDS: adhesion, cohesion, ductility, roofing, rubberized asphalt, toughness-tenacity test

Asphalts have been used for on-site preparation of built-up roofing for many years, but in recent decades there have been reports of increasing numbers of failures. One of the causes is the manner in which asphalt responds to extremes in temperature. If it is hard so that it will not flow when heated by the summer sun, it will often be brittle and crack badly at low temperatures. Conversely, asphalts that do not become too brittle in winter generally flow excessively in summer, particularly on a steeply sloped roof. To overcome such problems, rubber is sometimes added to the asphalt.

It was probably the rubberlike character of many bituminous materials that gave rise to the concept of adding rubber to asphalts, tars, and pitches. For well over 100 years, rubber has been included in bituminous materials to impart elasticity and improve serviceability in such products as caulks, mastics, and sealants. This was followed by extensive use of rubberized asphalt in road pavements. Indeed, most rubber manufacturers, asphalt producers, and highway departments of industrialized countries have been involved in the development and application of rubberized asphalts [1]. Numerous companies market a variety of such proprietary products, generally of undisclosed composition, including roofing materials for hot application.

Blending small quantities of natural, synthetic, or reclaimed rubber with asphalt causes major changes in its physical properties; for example, in viscosity or flow, extensibility, and brittleness. The stretchable nature of rubber restricts the propagation of microcracks that may appear because of impact loading, thus increasing the low-temperature impact resistance of the blend. Some

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researchers have claimed improvements in adhesion and resistance to weathering by the addition of rubber [2].

The characterization of the different rubberized asphalts is much more difficult than that of the various types of rubber, since the elastic properties vary considerably with the viscosity of the blend. Smith [2] infers from viscosity and extensibility test results that the improved properties require molecular (or near-molecular) dispersion of rubber in the asphalt. Larger rubber particles that may be present appear to make little contribution to an increase in viscosity, but reduce extensibility due to the setting up of stress concentrations. Smith's conclusion is contrary to that of Ritchie [3] in his discussion of the role of rubbery polymer addition to brittle polyvinyl chloride (PVC). Ritchie states that the larger the number of particles (or the smaller the size of the particles for a given percentage of rubber), the better the overall reinforcing action, although completely soluble rubbers that disperse on the molecular scale have little or no toughening effect.

A test method for measuring adhesion and cohesion properties of rubberized asphalt was developed by Benson [4] as early as 1955. It was based on the area under the load-elongation curve obtained by pulling a hemispherical head out of a sample of asphalt that had been poured hot into a cup and then cooled, and was called the "toughness-tenacity test." Although the name was selected within the context of the test, it may not be technically correct. Benson's equipment and test conditions have been examined and modified by various researchers since that time. In one important modification by Laaly, the size of the cup was increased and the hemispherical head was replaced by a threaded rod to improve the holding capacity during pulling. This modified procedure was included in the Canadian General Standards Board (CGSB) standard for hot-applied rubberized asphalt.³ The cup assembly and the various heads now reported are shown in Figs. 1 and 2, respectively.

In spite of extensive studies by Melley [5], among others, the order of toughness was the reverse of that expected when the modified procedure was applied to several rubberized asphalts and three unmodified asphalts. In another case, the same rubberized asphalt was tested by four laboratories and interlaboratory agreement was so poor that two laboratories found the material complied with a standard while the other two found that it did not. When the material was retested at the Institute for Research in Construction, National Research Council of Canada, on two additional days the within-laboratory precision for the maximum force was found to be rather poor (Table 1). The maximum acceptable range [ASTM Practice for Interlaboratory Testing of Paint and Related Materials (D 3980-83)] of means for the three days was 225 N.

As a result, the authors decided to study the measurement of toughness, adhesion, and cohesion of rubberized asphalts. The effects of the modification of "pull-heads" and test conditions are reported and test results are compared with those of the ductility test and the straight pull-off adhesion test.

Experimental Procedure

Materials

Rubberized asphalts from four manufacturers were selected at random. A sufficient quantity of each material was acquired from each source to permit all the testing to be carried out on the same batch of material. This was done to avoid any variation in materials resulting from minor changes in the formulation or processing conditions at the plant. The four materials are designated A, B, C, and D.

Toughness-Tenacity Test

The rubberized asphalt sample was heated in an oil-jacketed kettle to a temperature within the manufacturer's recommended temperature range. A sufficient quantity of molten material was

³ CGSB Standard 37-GP-50M, Hot-Applied Rubberized Asphalt for Roofing and Waterproofing, Canadian General Standards Board, Ottawa, March 1978.

FIG. 1—Cup assembly for toughness-tenacity test.

poured into six cups and the pull-heads were immersed and centered by means of a spider (Fig. 1) so that the top surface of asphalt rose to the specified level. Each assembly was allowed to cool at the standard temperature of $23 \pm 2^\circ\text{C}$ for 24 h. It was then mounted on the tensile tester and the head was pulled at selected speeds. In this way, graphs of the pull force against the head movement (elongation) for each speed were obtained. The four materials were subjected to

FIG. 2—Different pulling heads used in toughness-tenacity tests: (a) hemispherical head, (b) standard head, (c) tapering head.

TABLE 1—Toughness-tenacity results on Material C.^a

Test Day	Maximum Pull Force (F), N	Head Travel, mm	Toughness (T), N·m	T/F Ratio, mm
First day	589	94	21.4	36
	569	99	24.3	43
	402 ^b	215 ^c	43 ^c	107 ^c
$\bar{X}$	520		29.6	62
Revised $\bar{X}$	579	96.5	22.8	39.5
Sixth day	582	91	22.3	38
	667	93	25.0	37
	657	94	24.1	37
$\bar{X}$	635	92.7	23.8	37.5
26th day	607	101	25.1	41
	624	99	22.9	37
	598	100	25.7	45
$\bar{X}$	610	100	24.6	41

^a Conditions: standard head, 500 mm/min pulling speed.

^b Rejectable at the $P = 0.05$ significance level.

^c Rejectable at the $P = 0.01$ significance level using Dixon's test for extreme values, ASTM Practice D 3980-83.

the toughness-tenacity test at speeds of 10, 20, 50, 100, 200, or 500 mm/min. The last speed was used in most of the later tests because it is commonly used in the pull-off adhesion test (described below). The curves for Material C are shown in Fig. 3a.

It was observed that for most rubberized asphalts the peak load is obtained in the initial few millimetres of head movement when a mass of rubberized asphalt adhering to the sides and bottom of the head is pulled up with it. As the head rises further with the pull-force, the material on the bottom loses adhesion, causing separation of that portion. This, in turn, causes a drop in the pull-force and creates a void within the mass of rubberized asphalt (Fig. 4). Thereafter, a tubular mass of material with its upper end holding to the sides of the head keeps stretching and pulling more material from the cup (Fig. 5) until failure occurs in one of three modes:

- (a) rupture of the tubular section,
- (b) loss of grip or adhesion to the sides of the head, or
- (c) partial rupture and loss of grip.

Three modifications of the head were studied to determine which gave the clearest indication of adhesive or cohesive failure.

The initial pull-force and the quantity pulled from the cup depend on the properties of the rubberized asphalt. As the test proceeds, a portion of rubberized asphalt separates from the head owing to loss of adhesion to the bottom surface or the lowest portion of the head, whether it is a threaded rod or hemispherical head. To eliminate adhesion, the bottom of the head was covered with a thin film of a vegetable lecithin, which does not burn or smear the sides when the head is immersed in hot rubberized asphalt.

Second, to reduce or eliminate the area of the bottom surface of the head, a tapered one (Head C in Fig. 2) was designed, conforming in shape to a streamlined rod end being pulled from a highly viscous fluid. The tapering configuration reduced the bottom surface to a gradually narrowing

FIG. 3—Toughness-tenacity curves for Material C at various head-pulling speeds: (a) using a standard head (b) using a standard head with the bottom coated.

FIG. 4—Void within a mass of rubberized asphalt created by loss of adhesion to the bottom of the pull-head.

tip, and the head had its surface grooved with rings to improve retention of rubberized asphalt. The objective was to keep the stretching column of rubberized asphalt solid instead of hollow.

Third, a collar originally in the form of a 12.7-mm ($\frac{1}{2}$ -in.) hexagonal nut was added to the top of the standard head. While experimenting with various heads and configurations, it had been noticed that when the material covered the head the grip was better and failure tended to occur cohesively. Accordingly, a 6-mm-thick collar was mounted on top of the head (Figs. 1 and 2, Head *b*), and the quantity of hot rubberized asphalt poured into the test cup was increased to the level of the top of the collar. The test was carried out on all materials, with one pull at each of the six speeds, with and without the bottom surface of the head coated. The results for Material B are shown in Fig. 6. Finally, six specimens of each sample were tested at a speed of 500 mm/min with the collar on the top and coating on the bottom of the head. The results of all these tests are presented in Table 2.

In order to compare the results with those for the original hemispherical head developed by Benson [4], a few specimens of each material were tested with the standard head (Head *b* in Fig. 2) immersed to the top of the collar rather than the hemisphere (Table 3). A total of 178 specimens of the four samples were tested for toughness-tenacity at several speeds and with different heads.

FIG. 5—Progression of the toughness-tenacity test on rubberized asphalt.

FIG. 6—Toughness-tenacity curves for Material B, at various pulling speeds: (a) using a collar-modified standard head, (b) using a collar-modified and bottom-coated standard head.

TABLE 2—Toughness-tenacity tests.

Head Type	Material	Speed of Pulling, mm/min	Maximum Pull Force (F), N	Head Travel, mm	Toughness (T), N·m	T/F Ratio, mm	Type of Failure
Hemispherical	A ^a	100	101.0 ^d	75	5.5	54	partly adhesive
	B ^b	100	149.4 ^d	78	6.7	38	adhesive
	C ^a	100	96.7 ^d	75	4.9	52	adhesive
	D ^a	100	95.2 ^d	61	4.1	44	partly adhesive
Standard	A ^a	500	230 ^d	87	13.0	56	cohesive
	B ^c	500	315 ^d	38	7.4	24	adhesive
	C ^b	500	461 ^d	115	20.8	45	mostly adhesive
	D ^b	500	213 ^d	62	8.1	38	cohesive
Standard, bottom coated ^a	A	500	160 ^e	89	12.8	80	cohesive
	B	500	235 ^e	40	6.5	28	adhesive
	C	500	269 ^e	122	19.9	74	partly adhesive
	D	500	159 ^f	69	7.8	49	cohesive
Tapering ^a	A	500	201 ^d	61	9.3	46	adhesive
	B	500	235 ^d	45	7.4	32	adhesive
	C	500	338 ^d	60	9.7	29	adhesive
	D	500	231 ^d	63	9.3	40	mostly adhesive
Standard, with collar on top, bottom coated ^c	A	500	222 ^e	101	18.8	85	cohesive
	B	500	352 ^e	86	18.6	53	partly adhesive
	C	500	317 ^e	138	32.3	102	partly adhesive
	D	500	238 ^f	84	13.2	55	cohesive

^a Single value.^b Mean of three values.^c Mean of six values.^d First peak higher.^e Tenacity shoulder higher.^f Both peaks fairly similar.

TABLE 3—Comparison of hemispherical and standard heads.^a

Head	Material	No. of Tests	Pull Force, N		Head Travel, mm	Toughness, N · m
			Maximum	(Range)		
Hemispherical ^b						
	A	1	101	(. . .)	75	5.5
	B	3	149.4	(40.2)	77.7	7.04
	C	2	96.7	(2.9)	74.7	5.14
	D	2	95.2	(5.9)	61	4.1
Standard ^c						
	A	1	157	(. . .)	75	8.04
	B	1	201	(. . .)	50	6.08
	C	3	215.8	(88)	87.3	12.5
	D	3	158.6	(2.9)	55.3	5.8

^a Pulling speed = 100 mm/min.^b With collar.^c Without collar.

Pull-Off Adhesion Test

The pull-off test (Fig. 7) was used to evaluate the adhesive strength of rubberized asphalt. A 6-mm-thick layer was cast between a pair of brass cylinders each 38 mm in diameter. These had been cleaned and abraded with a fine emery cloth. After conditioning the assembly for 24 h at standard temperature and humidity, the cylinders were pulled apart. In the International Organization for Standardization (ISO) method for measuring adhesion of organic coatings,⁴ the tensile force is applied at such a rate that failure occurs within 90 s of the initial application. Consequently, a crosshead speed of 500 mm (20 in.)/min was used; this corresponds to the rate in the ASTM Test Methods for Rubber Properties in Tension (D 412-83) for tensile testing of rubber. By also applying the force at a slower rate, information on the viscoelastic properties of a material may be obtained. Overall, 54 specimens were tested for adhesion. The results are presented in Table 4 for brass surfaces only.

Ductility Test

This is a standard test method [ASTM Test for Ductility of Bituminous Materials (D 113-85)] for measuring the ductility of bituminous materials. In a preliminary test, one of the rubberized asphalts was tested at a speed of 50 mm/min at room temperature. It was observed that the material, highly stretchable at normal temperatures, was limp and would extend and detach itself from the mould clips. Accordingly, the method was modified by carrying out the test at a lower temperature using the common vertical tensile testing instrument. Each material was heated and poured into moulds that were then conditioned at -30°C for 24 h. Thereafter, each specimen was pulled apart at speeds of 50 or 100 mm/min at -30°C in an environmental chamber mounted on the tension testing machine. The experiment was repeated with specimens conditioned and tension tested at standard temperature (23 ± 2°C) and humidity (50 ± 5% relative humidity) at a speed of 100 mm/min to avoid pulling the material out of the mould clips. A total of 97 specimens were tested. The results are given in Tables 5a and 5b.

Results and Discussion

Toughness-Tenacity Test

The load-elongation curves obtained in testing Materials C and B with the standard head at different speeds are shown in Figs. 3a and 6a, respectively. It is evident that the area under the

⁴ ISO Standard 4624, Paints and Varnishes—Pull-Off Test for Adhesion.

FIG. 7—Adhesion pull-off test on a rubberized asphalt sample using a tension testing machine.

TABLE 4—Adhesion test results at 22°C.^a

Pulling Speed, mm/min	Material	Maximum Pull Force, N	Extension, mm	Failure Mode
10	A	115	5.5	adhesive
	B	129	7.1	adhesive
	C	132	6.8	adhesive
	D	155	32.5	cohesive
500	A	249	4.7	adhesive
	B	315	8.8	adhesive
	C	388	5.0	adhesive
	D	313	33.0	cohesive

^a Means of six values.

TABLE 5a—Ductility of rubberized asphalt at different temperatures.

Material	Speed, mm/min	22°C				-30°C					
		No. of Tests	Pull Force, N		Travel, mm		No. of Tests	Pull Force, N		Travel, mm	
			Maximum	(Range)	Distance	(Range)		Maximum	(Range)	Distance	(Range)
A	50	...	...	(0.9)	...	(8)	3	410	(19.5)	13.6	(1.0)
	100	4 ^a	8.7		336		3	526	(59)	10.5	(1.5)
B	50	6	7.1	(1.9)	281	(63)	2	330	(90)	36.5	(9)
	100	4	11.8	(1.0)	324	(35)	2	506	(47)	29.5	(11)
C	50	...	...	(2.6)	...	(12)	2	925	(54)	4.0	(1)
	100	5 ^b	14.1		380		6	820	(392)	5.6	(3.5)
D	50	...	...	(0.2)	...	(52)	3	361	(55)	9.6	(2.9)
	100	6	4.85		239		3	422	(105)	9.7	(0.5)

^a One result discarded because of slippage and one on the basis of Dixon's test.^b One result discarded on the basis of Dixon's test.

TABLE 5b—Changes in ductility with test conditions.

Material	Speed Change from 50 to 100 mm/min				Temperature Change from 22°C to -33°C			
	22°C		-30°C		50 mm/min		100 mm/min	
	Force, N	mm	Force, N	mm	Force, N	mm	Force, N	mm
A	...	...	+116	-3.1	...	...	+517	-327
B	+4.7	+43	+176	-7.0	+223	-244.5	+494	-294.5
C	...	...	-105	+1.6	...	...	+806	-374
D	...	...	+61	+0.1	...	...	+417	-229

curve (so-called "toughness") increases with the speed of loading. This is contrary to the known behavior of viscoelastic materials and the principles of mechanics, but it can be explained by the fact that the higher force at increased speed tends to pull more material out of the test cup, resulting in greater crosshead travel and, hence, in a larger area under the curve. It is apparent that a bigger cup and greater amount of rubberized asphalt would lead to a still larger area under the curve, up to a certain limit. This limit depends, of course, on the adhesive and cohesive properties of the material and the specimen being sufficiently large for the boundaries of the cup to have a negligible effect. Consequently, the method does not measure toughness; the higher values result from excessive elongation, the equivalent of jaw slippage in a tension test.

Treating the bottom of the head substantially reduced adhesion to it. This is quite clear from the manner of failure and from comparison of the curves in Figs. 3a and 6a with those in 3b and 6b, respectively. The loss of adhesion leaves a void in the mass, and the material being pulled is reduced to a rubbery tube with its top hanging on the sides of the head and the bottom end pulling more material from the cup (Fig. 4). Benson originally proposed that the area under the shoulder of the curve should be referred to as "tenacity," which is a measure of adhesion. Owing to the difficulty in differentiating the second area from the first, some users replaced this parameter with "ratio of total area to the first peak load," obtaining, in effect, a modified elongation result. This ratio was also included in CGSB Standard 37-GP-50M. When adhesion was deliberately reduced by coating the bottom of the head, however, the shoulder of the curve was not greatly affected, but the initial peak was reduced (Fig. 3b) or practically eliminated (Fig. 6b), which resulted in larger ratios for all four materials. It is evident that neither method of treating the results relates to adhesion.

To establish the effectiveness of the head adopted in CGSB Standard 37-GP-50M in relation to the original hemispherical head, it is necessary to compare results from tests conducted at the same speed. The values given in Table 3 show that the pulling force exerted by the standard head without the collar is larger for all four rubberized asphalts. The distance the head travels seems to be more affected by the material being tested than by the head geometry and is probably related to its adhesive and tensile properties. The toughness values are higher with the standard head in three of the four cases and, with Material C, significantly higher. As will be shown, this rubberized asphalt is the least extensible of the four. Because of the small number of tests performed at each speed, it was not possible to establish whether one head provides more consistent results. The curves obtained with the hemispherical head were less distinctive, however. For example, with Material A the initial peak was lower in relation to the remainder of the curve (Fig. 8), confirming that the head developed by Laaly is an improvement over the original.

In comparison with those for the standard head, the load-elongation curves obtained with the

FIG. 8—Toughness-tenacity curves for Materials A and D, using different heads at one pulling speed.

tapered head varied from being fairly similar for Material D to complete loss of the initial peak with Material A (Fig. 8). For the other two materials, the curves were more like the hemispherical curves. With three of the four rubberized asphalts, the peak load was higher for the tapered head than for the original head, but it was lower than for the standard head. During the tests, the same sequence of separation from the lower part of the head and thinning of the tube walls occurred as with the other two heads. In addition, the incidence of slippage from the top threads was much higher than it was with the standard head. Consequently, work with this design was terminated.

The use of a collar (a hexagonal nut in the present studies) with the bottom of the standard head coated with lecithin raised the maximum force values close to those for the uncoated standard head and made the distance the head traveled more uniform. The toughness values also increased and separated the materials into three groups: in descending order, Materials C, A and B, and D. The ratio of toughness to force that was used in place of the original tenacity calculation also placed Material C at the top of the group. These results will be compared in the next section with the separate measurements of adhesion and ductility.

Adhesion

The pull-off test results given in Table 4 refer to adhesion to brass surfaces only. A similar set-up can be used for other surfaces, such as concrete, steel, wood, etc. The mode of failure for Materials A, B, and C indicates that these materials were pulled clean off the brass surface, which suggests that for rough and porous surfaces the pull force or force of adhesion could be much higher.

It is clear that Material D has much better adhesion than the other three. It is equally obvious that the only result from the previous tests that agrees with this finding is the qualitative observation that Material D never failed strictly in adhesion. Thus, if tenacity is equated with adhesion, the previous test does not rate the materials in the correct order.

In addition, Material D is the only one of the four to exhibit high tensile strength and extension (elongation), both of which are required for toughness. Loss of adhesion by the other three materials, however, probably resulted in low extensibility, as measured by the test. As expected, the increase in loading rate of 50 times resulted in marked increases in the maximum force at which failure occurred. Materials B and D did not exhibit a corresponding decrease in extension, while Materials A and C did, indicating a tendency to brittle behavior at high rates of stress application or at low temperatures (time-temperature inverse relation). Again, these trends are not evident in the results presented in Table 2.

Ductility

ASTM Test D 113 was developed for unmodified asphalt, a relatively brittle material. This, of course, is why asphalt is modified with rubber. Asphalt is normally tested at a temperature of $25 \pm 0.5^\circ\text{C}$ ($77 \pm 1^\circ\text{F}$) and speed of 50 mm/min. The requirement for ductility at 25°C in the ASTM Specification for Asphalt Used in Roofing (D 312-84) ranges from 100 mm for Type I asphalt to 15 mm for Type IV and in the ASTM Specification for Asphalt Used in Dampproofing and Waterproofing [D 449-79 (1983)] from 300 mm for Type I to 20 mm for Type III. Consequently, except for Type I asphalt in Specification D 449, lowering the temperature or increasing the test rate could only slightly reduce the extension at which the asphalt specimen ruptures.

With the more viscoelastic rubberized asphalt the results for both tensile strength, as reflected in the force at which failure occurs, and elongation at failure (Table 5a) can be considered together without concern for the effect of adhesion present in the pull-off test. At the standard temperature and a speed of 100 mm/min, Material C requires the greatest force but extends the most. Material D is the lowest in both parameters, although the levels of force for all four materials are relatively low. At the lower temperature at both speeds, however, Material C exhibits much higher loading and lower extensibility, again indicating more brittleness. With the other three the force levels are about the same, within experimental error, while Material B has the greatest extensibility. Unfortunately, the low-temperature force results for Material C at 100 mm/min are less reliable than the others because of the large range, which is statistically different at the significance level of $P = 0.01$. None of the individual readings could be discarded because all were uniformly distributed over the range.

With rubberized asphalt, it is more informative to compare the reaction of the different materials to changes in the test rate or test temperature than to compare the results at one test condition (Table 5b). Because of the small difference in the two rates, changing the temperature has a much greater effect, especially on reducing extensibility. To produce a comparable difference by changing speed would probably require the second speed to be at least 500 mm/min. With the change in temperature at 100 mm/min, Material D exhibited the smallest changes in force and ductility and, with the change in rate at -30°C , the smallest change in ductility. When subjected to a temperature decrease, it follows that Material D has less tendency to brittleness. Material C, on the other hand, exhibits the greatest change with temperature change; as noted, the change-in-force results produced by the change in rates are questionable. Materials A and B are fairly comparable, with A reacting less to rate change and B tending to react less to temperature change. Hence, this test places the materials in the descending order of D, A and B, and C, the exact opposite of the results for the so-called toughness-tenacity test.

Summary and Conclusions

Changing the shape of the pull-head from hemispherical to a threaded rod with a gently rounded bottom (standard head) improved the ability to distinguish between different rubberized asphalts,

particularly when comparing load-elongation curves (Fig. 8). Tapering the threaded rod increased slippage from the upper threads, showing that this design is intermediate in shape and performance between the previous heads. Placing a collar on the top of the standard head and raising the level of rubberized asphalt to the top of the collar reduced slippage, so that the results of the test were less affected by adhesion of the material to the threads.

Load-elongation curves for pull-out tests conducted at different speeds and with various heads yield larger areas, supposedly a measure of toughness, with increased speed of loading. Viscoelastic materials, however, are known to become brittle (less tough) at higher test rates (and lower temperatures). In this case, the areas increase with higher speed because more material is pulled from the cup, allowing greater crosshead movement before failure occurs. As a result, the method does not specifically determine toughness, although this property may have some effect on the values obtained.

In an attempt to distinguish between the adhesive and cohesive elements in the pull-out test, the influence of adhesion to the rounded bottom of the threaded rod was eliminated by coating it with vegetable lecithin. The result was that the secondary area of the curve, which is supposed to quantify tenacity, was little changed, while the initial peak was greatly reduced. Thus, the ratio of toughness (T) to maximum pull force (F), T/F , increased instead of decreasing. Evidently, the method does not evaluate adhesive properties. When, simultaneously, the collar was placed on top of the standard head and the bottom was treated with lecithin, the head travel increased because adhesion was reduced and the toughness values were increased. On this basis, the materials would be rated as C, highest; A and B, intermediate; and D, lowest.

One objective of this work was to determine whether the results of the toughness-tenacity test agree with separate measurements of adhesion (related to tenacity) and cohesion or toughness (a combination of the properties of tensile strength and elongation). The pull-off test showed that Material D has the best adhesion since it always failed cohesively. This superiority could be detected by the pull-out test only from qualitative assessment of the failure type. In the adhesion study, Material D was the only one to have both high tensile strength and extensibility, although the latter property may have been underestimated for the other three because of their failure in adhesion. Increasing the loading rate of the test produced a better retention of extensibility for Materials D and B.

When the ductility test is performed, interference caused by the level of adhesion is eliminated through the design of the clips that pull the specimen. Consequently, a better evaluation of the tensile properties can be made. When the test was carried out at -30°C , Material C exhibited much higher loading and lower extensibility, a combination that is equated with brittleness. When the changes in properties with changes in test temperature are examined, Material D is the toughest. Consequently, the ranking is in the following order: D, A and B, and C. When both adhesion and ductility results are considered together, the order of performance is $D \gg B > A \gg C$.

The reason for the change in the order produced by the pull-out test is that it was designed to include all three properties of importance: tensile strength, elongation, and adhesion. However, studies at the Institute for Research in Construction have established an important principle in the development of evaluation methods; that is, if several properties are involved in a particular service it is better to measure them separately than to try to incorporate them all in one test [6]. The combination of properties in tests that are basically empirical often makes it difficult to isolate important factors or to distinguish levels of quality; this combination sometimes leads to results that do not agree with known behavior or performance and invariably makes the method imprecise. Even when within-laboratory precision is improved as a result of careful study, interlaboratory agreement remains unsatisfactory. Examples of this include tests for measuring abrasion resistance, scratch adhesion, and scrubability of organic coatings, where only the order of ranking should be used for comparing results obtained in different laboratories. To conclude, with rubberized

asphalts and other materials intended for the same use, it is preferable to measure adhesion and tensile properties separately.

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