Effect of Fillers on the Marshall Stability Of Bituminous Mixtures

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This paper presents the results of experiments which show the large effect of filler quantity and filler type on the Marshall stability of bituminous concrete. The mathematical relationship of filler (quantity times type) to the stability of an asphaltic concrete mixture using sand-gravel aggregate is shown, and a simple test described by which the quantitative effect of various types of fillers on stability may be determined.

•THE LARGE EFFECT of variations in quantity of filler on the Marshall stability of asphaltic concrete mixtures is well known (1 through 5). In 1958, for instance, the authors showed (6) that the stability of a sand-gravel asphaltic concrete (NABC) was raised from 700 to 2,100 lb by increasing the quantity of commercial limestone filler in the mix from 2 to 10 percent by weight. It has also been amply demonstrated that equal quantities of different types of fillers have different effects on the properties of both filler-asphalt mortars (4, 7) and on the Marshall stability of paving mixtures (8, 9).

It would be desirable to have a way of separating and measuring the relative importance of the effects of quantity and the effects of quality of filler on some common parameter, such as Marshall stability. However, a test method that would give an index of quality or effectiveness of filler based on primary properties has been traditionally elusive. In spite of much work, previous efforts to find such a test have been generally unsuccessful. Various empirical methods largely related to soils technology have been used for specification and control purposes; for example, (7, 10). After extensive research the best answer known to date remains that stated in 1937 by Traxler (11):

> From a practical point of view it is evident that if we want to know how a filler will affect an asphalt, the best way to obtain such information is to mix the asphalt and mineral powder and compare the properties of the mixture with that of the asphalt.

There have been numerous attempts (e.g., 1 through 5) to relate one form or another of bulk density to filler performance. Although a broad directional trend is well recognized, the relationship has not been generally useful. The authors' studies (7), reported in 1959, also failed to confirm the bulk density correlation over a wide range, but did show that the viscosity characteristics of the mortar, as measured by penetration, softening point, and ductility, correlate well with Marshall stability and with known service behavior of various fillers in asphaltic concrete pavements.

Mitchell and Lee (12) showed a correlation between the bulk volume of various fillers and the viscosities of both filler-tar and filler-asphalt mortars. They determined their bulk volume by boiling a suspension of filler material in benzine or a similar liquid to remove entrapped air, and then measuring settled volume. The weight of the settled material was divided by its volume to obtain a bulk density number. This method introduces other factors (wetting by, and settlement in, an organic liquid) that are not encountered in the normal determination of bulk density by dry compaction. The bulking concept as measured by settlement rate has, of course, demonstrated its merit in other areas. The Hveem sand equivalent test (13), for instance, is a good illustration

of the general principle, but it involves induced flocculation in an aqueous medium.

The Mitchell and Lee findings in particular led back to reconsideration of the influence of the medium on bulking characteristics. The behavior of high surface area particles depends not only on their physical and chemical characteristics and their concentration but also depends on the nature of their surroundings or the media to which they are exposed. Inasmuch as paving fillers are for use in a bituminous mixture, something might be learned by applying the bulking concept in petroleum media. With this background for inspiration and as a logical continuation of the authors' long-range studies of fillers, it was therefore decided to explore further the possibility of a bulking factor in a petroleum hydrocarbon as an index of filler quality.

The liquid chosen was kerosene. The procedure is detailed in the Appendix. It consists essentially of boiling 10 g of dry filler in approximately 50 ml of kerosene, making total volume up to 100 ml with kerosene, then allowing to settle for 24 hr. The volume of settled filler is read as the "bulk volume." This bulk volume is then related to the volume that would be occupied by an equal weight of solid filler material. This is called the "solid volume" and it is obtained by dividing the weight of material used (10 g) by the specific gravity. The ratio of the bulk volume to the solid volume is called activity coefficient K. It is postulated that this parameter might reflect a combination of those elements of particle-size distribution, wetting and bulking in petroleum, plus factors still unknown, which are pertinent as a measure of filler quality in asphaltic concrete mixtures.

Mathematically

$$K = \frac{\text{bulk volume}}{\text{solid volume}}$$

which, using the suggested procedure, becomes

$$\frac{\text{read volume of settled filler in ml}}{\text{weight of sample (10 g)}} \text{ times specific gravity.}$$

RESULTS

The activity coefficient K as determined by this method is given in Table 1 for some common fillers. Generally, the K-values range from about 2 to about 11. These values are dependent on the test procedure used. A different solvent, different equipment, or a change in settling time would, of course, result in different numbers. However, it is their relative values, rather than the testing details, that are currently under preliminary study as a means of measuring relative filler activity. There is no intention at this time to attempt correlation with primary filler properties. Therefore, detailed physical data on most of these fillers are not included in the paper.

The relation of K to particle size for a given single filler type is illustrated by the data given in Table 2. Here four samples of ground silica having the same chemical analysis, but of different particle sizes, (surface area range from 8,386 to 20,600 sq cm per g) are related to K (range 3.8 to 9.2). As would be expected from Stoke's Law, K, under these conditions, proved to be inversely proportional to average particle size and directly proportional to mean surface area over the range studied. Marshall stability of standard mixes made with equal quantities of these four silica fillers showed good correlation with both K and with the normal particle-size or surface area parameters. The case for different particle sizes of the same filler material would thus seem to be comparatively simple and straightforward. Even here, however, the activity coefficient K might have worthwhile application because of relative ease of testing.

Unfortunately different fillers can impart widely different properties to bituminous mixes that cannot be explained by or correlated with dimensional factors alone. Further, ordinary bulk density correlations have not been satisfactory over an acceptable range, nor have numerous attempts to relate behavior in a mix to other filler properties been successful. Thus, though particle size and shape are undoubtedly important considerations, there are other forces that influence filler activity or quality. It is this combination of dimensional and "other" factors that needs to be pinned down and measured as an index of filler quality. If K should prove to be a means of tying together the effects on Marshall stability of a number of different filler types when used

TABLE 1

VALUES OF ACTIVITY COEFFICIENT K FOR VARIOUS FILLERS

Filler	Specific Gravity	Bulk Vol. (ml/10 g)	K
Lake sand	2 601	7 1	1 8
Limestone	2 801	7.5	2.1
Wheat flour	1 436	15.4	2.2
Fly ash	2.475	12.0	3.0
Aluminum silicate	2.588	24 4	6.3
Asbestine	2.780	23.8	6.6
Ground silica (μ)			
30	2.636	14 3	3.8
15	2.639	22 0	5.8
10	2.649	26.0	6.9
5	2.615	35.0	9.2
Hydrated lime	2 463	27.2	6.7
Magnesia·			
200 mesh	3.102	24.8	7.7
325 mesh	2.775	34.9	9.7
Kaolin clay	2.553	42.9	11.0
Asbestos, 7M	2 617	50.0	13.1

in concentrations covering the practical working range, this might be a correct lead.

The five fillers underlined in Table 1, having well-separated K-values, were selected for use in the Marshall stability test series. Both basic and acidic types of materials ordinarily used as fillers are represented. They were used at two concentration levels in a standard sand-gravel mix. This is a relatively noncritical, 3/8-in. maximum size, well-graded mix, which the authors have used for a number of different studies and which is described in detail in the literature (14). A single asphalt content, which had previously been found to be near optimum, was used for all specimens. Because this is a noncritical mix, deviations due to asphalt content adjustment are not considered significant for purposes of this paper. The making and testing of the Marshall specimens was in accordance with ASTM Designation D 1559-60T. The two levels of filler concentration used in these experiments were 0.55 and 0.30 filler to bitumen ratio by volume, $\frac{V_F}{V_B}$. These ratios are roughly equivalent to 7 and 4 percent filler by weight of total mix. All granular fillers were screened through the 200-mesh sieve before being weighed into the mix.

TABLE 2

RELATION OF ACTIVITY COEFFICIENT K TO PARTICLE SIZE, GROUND SILICA[1]

Property	Cumulative % Finer Than			
	30μ	15μ	10μ	5μ
Particle size (μ):				
40	99.7			
30	98.2			
20	90.5	99.7		
15	79.8	98.2		
12			99.7	
10	61.8	90.0	98.7	
6				99.6
5	35.6	60.0	80.0	98.1
4				94.0
3	23.7	40.0	57.0	85.5
2		28.5	40.0	68.0
1				36.0
Average particle size (surface mean) (μ)	2.7	2.0	1.6	1.1
Surface area (sq cm/g)	8,386	11,300	14,100	20,600
Activity coefficient K	3.8	5.8	6.9	9.2
Marshall stability (lb)	1,210	1,360	1,440	1,640

[1] From "Minusil" Pennsylvania Pulverizing Company. Chemical analysis (%): SiO_2, 99.2; Fe_2O_3, 0.016; Al_2O_3, 0.04; TiO_2, 0.008; CaO, trace; MgO, trace; loss on ign., 0.09.

TABLE 3

RELATION OF ACTIVITY COEFFICIENT K TO MARSHALL STABILITY

Filler	Identification	K	$\frac{V_F}{V_B}$	$\frac{W_F}{W_B}$	Calculated Stability[1]	Marshall Test Results Stability	Marshall Test Results Flow
Commercial limestone	A_1		0.30	0.82	771	730	8
	A_2	2.1	0.55	1.50	1,159	1,085	10
Ground silica 30μ	B_1		0.30	0.77	767	740	7
	B_2	3.8	0.55	1.41	1,241	1,210	9
Hydrated lime	C_1		0.30	0.72	758	795	8
	C_2	6.7	0.55	1.32	1,341	1,405	10
Ground silica 5μ	D_1		0.30	0.76	833	825	8
	D_2	9.2	0.55	1.39	1,639	1,640	11
Clay (kaolin)	E_1		0.30	0.74	828	825	8
	E_2	11.0	0.55	1.36	1,733	1,735	11

[1] $y = 660 + 250\,(0.4\,\frac{W_F}{W_B} - 0.25)\,(K + 3.6)$.

Marshall test results are given in Table 3. Marshall flow varies with filler concentration but is essentially independent of K. There is, however, a linear relationship between the K-value and Marshall stability at both levels of concentration, as shown in Figure 1. When these data were analyzed, it was found that the regression equation relating the filler-bitumen ratio and K with Marshall stability could be reduced to

$$\text{Marshall Stability} = 660 + 250(\frac{V_F}{V_B} - 0.25)(K + 3.6)$$

This equation contains a concentration factor $\frac{V_F}{V_B}$ and an independent "quality" factor K. For the benefit of those who might still find it easier to visualize filler concentration by weight, an approximation of this equation in terms of filler-bitumen ratio by weight is

$$\text{Marshall Stability} = 660 + 250(0.4\,\frac{W_F}{W_B} - 0.25)(K + 3.6)$$

(This approximation is based on assuming an average asphalt gravity of 1.03 and an average filler gravity of 2.6).

The weight-basis equation is used herein to facilitate illustration of the principles of this approach for evaluating filler activity. Though suitable for purposes of this preliminary report, the more precise and technically sound volume basis should be used for testing the relationship over a broader range of fillers and mixes.

Figure 2 shows the comparison of calculated stability values using the weight-basis formula vs the actual test results. Even using the approximate equation, the correlation is excellent and brackets the 45° diagonal line. This line represents data obtained

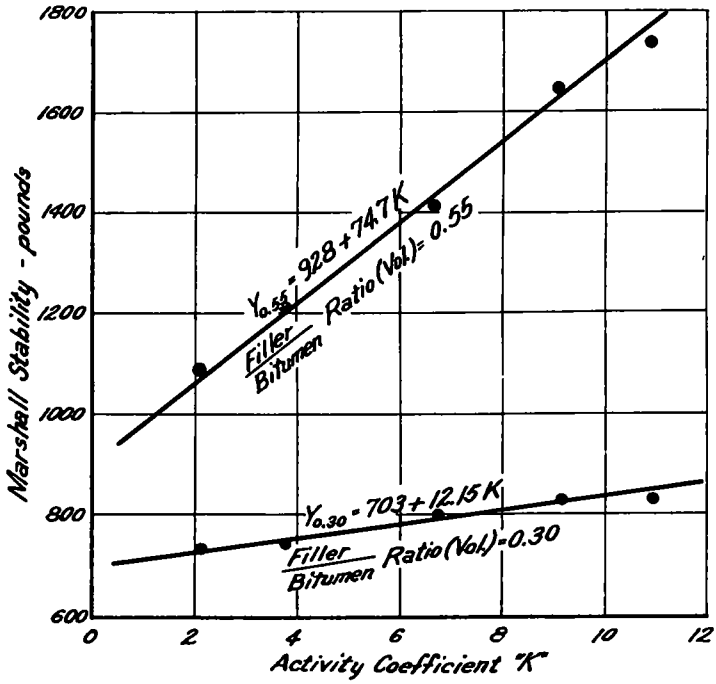

Figure 1. Marshall stability vs activity coefficient.

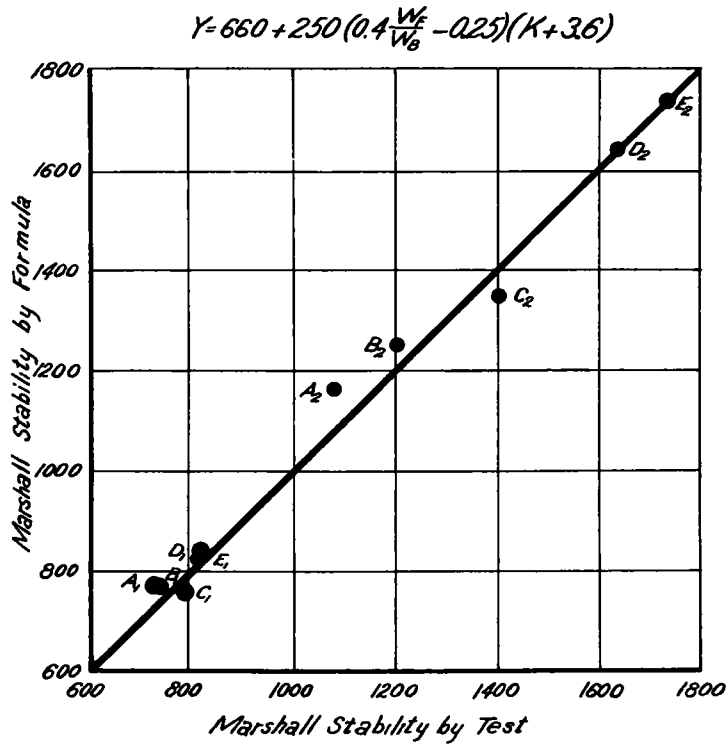

Figure 2. Comparison of calculated vs test values, Marshall stability.

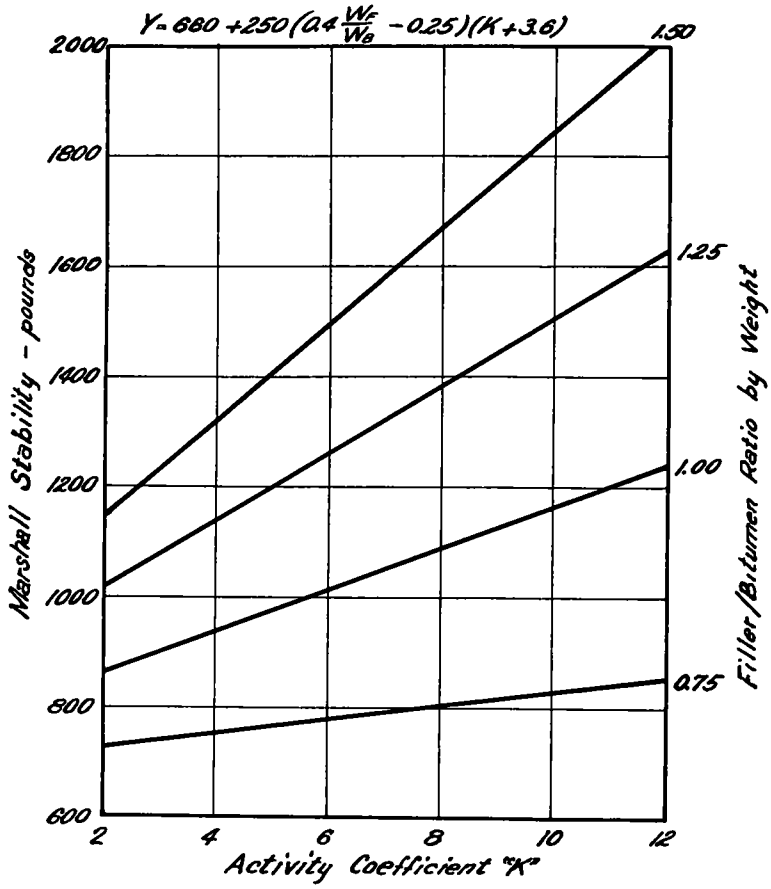

Figure 3. Effect of filler quantity times quality on Marshall stability.

at two different concentration levels, using five different fillers of widely varying physical and chemical properties. Thus, it would appear that at last, there is a potentially valid means of separating the effects of quantity and the effects of quality.

Figure 3 presents a way of combining these relationships to show graphically the effects of both quality at different concentration levels, and the effects of quantity for any given quality of filler. It is evident that quality differences (between K of 2 and 11, for instance) are not as significant at the low concentration ($\frac{W_F}{W_B}$ of 0.75) as they are at higher concentrations of filler. Conversely, analysis along the vertical scale shows that the concentration factor is not as critical with a low K-filler, such as limestone (K = 2.1), as it is with clay (K = 11.0). Although these general directions for individual types of fillers have, of course, been known for some time, there is now means of separating these factors for better visualization of interrelations and for studying both quality and quantity influences.

CONCLUSIONS

The following conclusions are based on observations of the behavior of a limited number of granular fillers used in a sand-gravel mix only. Although a somewhat broader range of fillers and concentrations than are reported herein have been checked out, no attempt, has been made as yet to test these relationships over a wide range of materials or mixes. The purpose and scope of this paper is limited to a preliminary

report of an approach to evaluating filler activity and for mathematically and graphically expressing the effects of filler concentration and filler type or activity on Marshall stability for a given bituminous concrete mix. At this stage, only enough experimental data are presented to illustrate the methods and technique. It is hoped that others will also find these concepts to be of interest and will join in further evaluating the potentials of the approach.

1. Marshall stability is a function of both filler concentration and filler type or activity. Differences in effect on stability due to filler activity increase as the concentration level is increased.
2. Marshall flow at constant asphalt content for a sand-gravel mix is a function of filler concentration but essentially independent of filler type over the range studied.
3. The ratio (K) of the bulk volume, as determined by the described method, to the solid volume of various inert, granular fillers, is an index of their relative effect on Marshall stability.
4. For a given filler type (ground silica), the activity coefficient K is directly proportional to mean surface area over the range of 8,386 to 20,600 sq cm per g.
5. For a given bituminous concrete mix, the effects on stability of changes in filler concentration and filler type or activity (as measured by K) may be conveniently expressed both mathematically and graphically, using techniques described.

Appendix

DETERMINING ACTIVITY COEFFICIENT OF MINERAL FILLERS

Purpose

To determine an activity coefficient K that will indicate the relative effect of various inert, granular, filler materials on filler-asphalt mortars and on the stability of asphalt paving mixtures.

Equipment

Pyrex 100 ml graduated cylinders (Thomas 4409-F).
Pyrex 100 ml volumetric flask.
Balance, sensitive to 0.01 g.
Stirring rod approximately 12 in. long.
Bunsen burner and associated equipment.

Method

1. Oven dry approximately 100 g of filler material to constant weight at 100 C and cool in desiccator.
2. Remove lumps by sieving. Only that portion of granular filler materials passing the No. 200 sieve is used in the following tests.
3. Determine specific gravity in accordance with ASTM D 854-52 using kerosene option and removing air by boiling.
4. Weigh 10 g of dry filler into graduated flask. Add approximately 50 ml of kerosene.
5. Place graduated cylinder over suitable heat source and boil gently for 10 min. Stir initially to keep filler in suspension until boiling starts.
6. At end of boiling period remove cylinder from heat, place in support, and fill to 100-ml mark with kerosene.
7. Stir until all filler is in suspension, then let stand undisturbed for 24 hr.
8. At end of 24-hr period, mix settled filler gently with stirring rod to break up stratification, but do not stir filler into suspension.

9. Read and record level of settled filler; this is the bulk volume.
10. Compute activity coefficient K by dividing bulk volume by solid volume of filler, or

$$K = \frac{\text{bulk volume}}{10\ g} \text{ times specific gravity}$$

REFERENCES

1. Traxler, R. N., Olmstead, F. R., and Bollen, R. "Comparison of the Various Tests Used to Evaluate Mineral Filler." Proc., AAPT, 9:218 (1937).
2. Traxler, R. N., and Miller, J. S., "Mineral Powders, Their Physical Properties and Stabilizing Effects." Proc., AAPT, 7:112 (1936).
3. Traxler, R. N., "The Evaluation of Mineral Powders as Fillers for Asphalts." Proc., AAPT, 8:60 (1937).
4. Evans, A., "The Physical Properties of Mixtures of Bitumen and Finely Divided Mineral Matter." Inst. of Petroleum Technical Jour., 18:No. 110, p. 957 (1932).
5. Fink, D. F., and Lettier, J. A., "Viscosity Effects in the Marshall Stability Test." Proc., AAPT, 20:246 (1951).
6. Hudson, S. B., "Practical Gradation Limits for Natural Aggregate Bituminous Concrete." M-W Tech. Paper 57, January 1958; Roads and Streets, 101:No. 5 and 6 (May and June 1958).
7. Warden, W. B., and Hudson, S. B., and Howell, H. C., "Evaluation of Mineral Fillers in Terms of Pavement Performance." Proc., AAPT, 28:316-346 (1959).
8. Kallas, B. F., and Puzinauskas, V. P., "A Study of Mineral Fillers in Asphalt Paving Mixtures." Proc., AAPT, Vol. 30 (1961).
9. "Investigation of the Design and Control of Asphalt Paving Mixtures." Corps of Engineers, U. S. Army, Tech. Memo. 3-254, Vols. 1, 2, and 3. Waterways Experiment Station, Vicksburg, Miss. (1948).
10. Bollen, R., "The Selection and Use of Mineral Fillers for Low Cost Roads." Proc., AAPT, 8:3 (1937).
11. Traxler, R. N., "The Evaluation of Pulverulent Solids as Fillers for Asphalts." Final Research Report, Barber Company, Inc. (Aug. 2, 1937).
12. Mitchell, J. G., and Lee, A. R., "The Evaluation of Fillers for Tar and Other Bituminous Surfacings." Jour. Soc. of Chemical Industry, 58:299-306 (1939).
13. Hveem, F. N., "Sand-Equivalent Test for Control of Materials During Construction." HRB Proc., 32:238-250 (1953).
14. Vokac, R., "Repeatability of the Marshall Test by Analysis of Factorial Experiment Data." Annual Meeting, AAPT (1962).