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Laboratory Tests for Selecting Wetting Agents for Coal Dust Control

By H. William Zeller



UNITED STATES DEPARTMENT OF THE INTERIOR

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

cm	centimeter	mL	milliliter
cm/s	centimeter per second	mm	millimeter
cP	centipoise	mM/L	millimole per liter
deg	degree	μm	micrometer
dyn/cm	dyne per centimeter	μmho/cm	micromho per centimeter
g	gram	pct	percent
h	hour	s	second
mg/m ³	milligram per cubic meter	wt pct	weight percent
min	minute		

LABORATORY TESTS FOR SELECTING WETTING AGENTS FOR COAL DUST CONTROL

By H. William Zeller¹

ABSTRACT

The Bureau of Mines is conducting research to determine whether the use of chemical surfactants improves respirable coal dust control and is evaluating laboratory test criteria for selecting effective surfactants. This publication presents the results from wetting effectiveness tests with emphasis on the capillary rise test.

The test variables investigated included coal particle size, type of surfactant, surfactant concentration, test duration, and mineral content of the water. In addition to the rise test, measurements of contact angle, zeta potential, and sink time for various coal and surfactant combinations were conducted. No significant correlations among the four wetting tests were observed, a surprising result which implies that each test type measures specific aspects of the wetting phenomenon and also that each test type should only be used to select surfactants for specific applications. For example, the rise test shows solution penetration into porous materials, and the sink test discriminates among surfactants for dispersing finely ground materials into suspension. Another important conclusion is that combinations of agents retain their individual wetting properties, allowing mixtures to be formulated to perform well in both rise and sink tests.

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INTRODUCTION

A serious, continuing problem in underground coal mining is the incidence of coal workers' pneumoconiosis, or black lung, caused by inhaled coal dust. The Federal Coal Mine Health and Safety Act of 1969 (Public Law 91-173) required strict compliance with the 2.0-mg/m³ standard for respirable coal dust by December 30, 1972. Although mines are now much less dusty than formerly (27),² problems in achieving compliance still exist in some mines, particularly those with longwalls (20).

Because the effective use of water is essential for dust control in underground coal mines, the Bureau of Mines is conducting research on improved spray performance (6-7), on development of scrubbers (10-12), and on shrouded spray systems to direct dusty air away from miners (23). The Bureau's interest in wetting agents³ began in 1940 with a publication (21) that suggested applications for wetting agents and presented results of limited laboratory tests with wetting agents on roadways. In 1963 (2), the Bureau measured dust loadings from continuous mining machines with and without the agents. Compared with water alone, additional dust reductions of about 30 pct were measured using water containing wetting agents. In 1974 (4), the Bureau tested wetting agents in a longwall experiment but obtained inconclusive results. In two reviews (1, 24) of mining dust control, the Bureau reported that mine operators obtained inconsistent results: The agents produced noticeable dust reduction in some mines but were ineffective in others.

²Underlined numbers in parentheses refer to items in the list of references preceding the appendix at the end of this report.

³Surfactants, or surface-active agents, are a family of substances which include detergents, emulsifiers, foamers, dispersants, and wetting agents. In this report the terms "wetting agent" and "surfactant" are used interchangeably.

In one of the few investigations involving both laboratory and underground tests of surfactants, Hargraves (19) also obtained inconclusive results. Two surfactants reduced dust 25 to 30 pct, but a third, the one that did best in the laboratory, produced no significant dust reduction underground.

Although numerous laboratory studies on coal wetting have been reported (5, 15-17, 28), none of these involved in-mine measurements, so the validity of the laboratory tests for predicting dust suppression potential had not been established.

Prompted by these confusing findings, the Bureau's current investigation has the following goals:

1. Characterize the interaction of coals with aqueous surfactant solutions in the laboratory.
2. Develop laboratory test criteria for selecting effective surfactants.
3. Determine whether or not surfactants significantly reduce dust in coal mines.
4. Verify whether laboratory tests predict dust control effectiveness.
5. Identify specific areas in mines where the surfactants are cost effective.

Progress has been made on all tasks except for establishing a positive correlation between laboratory and in-mine results. This publication presents the results of a laboratory program to evaluate wetting effectiveness tests and to characterize the behavior of aqueous surfactant solutions and coal dust. Another study (25) done by a Bureau contractor describes a combined laboratory and in-mine test program. The contractor found that surfactants reduced dust compared with water alone. (Water containing surfactants reduced respirable dust by 27

pct and total airborne dust by 36 pct.) No correlation was established between the in-mine results and the laboratory

test data. Application areas are noted throughout this report, but cost effectiveness has not been established.

ACKNOWLEDGMENT

The author thanks James R. Blair, mining engineering technician, of the Bureau's Twin Cities Research Center, for his assistance in developing some of the

test procedures, for conducting many of the laboratory tests reported in this publication, and for monitoring work conducted by Bureau contractors.

PROCEDURES

Four types of tests or measurements were conducted to determine the effectiveness of aqueous surfactant solutions for wetting coal. Only two of these tests, the capillary rise test and the sink test, were conducted by the Bureau. The measurements for contact angle and for zeta potential were done by contractors.

CAPILLARY RISE TEST

The capillary rise test was adapted from procedures recommended by Crawl and Wooldridge (8), who developed the technique for assessing the wettability of powders. In this method, the liquid, by capillary action, is allowed to rise unopposed in a column of powdered coal contained in a glass tube (fig. 1).

The glass tubes were 120 mm long with an internal diameter of 8 mm. A glass-fiber filter, 10 mm in diam, was attached to the tube's bottom with a water-insoluble glue, usually rubber cement. The tube was then filled with a measured quantity of minus 42-mesh coal. Consolidation of the coal column to a predetermined height was accomplished by lightly tapping the glass tube for 1 to 2 min. For most coal samples of 2 g, the final consolidated column height was about 60 mm.

The vertical tube of coal was then immersed in the test liquid so that the bottom of the coal column (top surface of the filter) was about 2 mm below the liquid surface. In most tests the tube of coal was removed from the test liquid

after a measured time interval and weighed, and the weight of absorbed liquid was calculated.

The rise rate was also often determined. In most tests this was done by recording the times when the liquid front reached specified column heights. For sufficiently slow rise rates, or for long tests, the tubes were removed from the liquid at specified intervals, weighed, and returned to the liquid. Another variation involved placing the container of the supply liquid alone on a balance and recording the change in weight as the liquid rose in the column. A Bureau contractor (14) tried a procedure in which the test liquid was introduced to the top of the coal column.

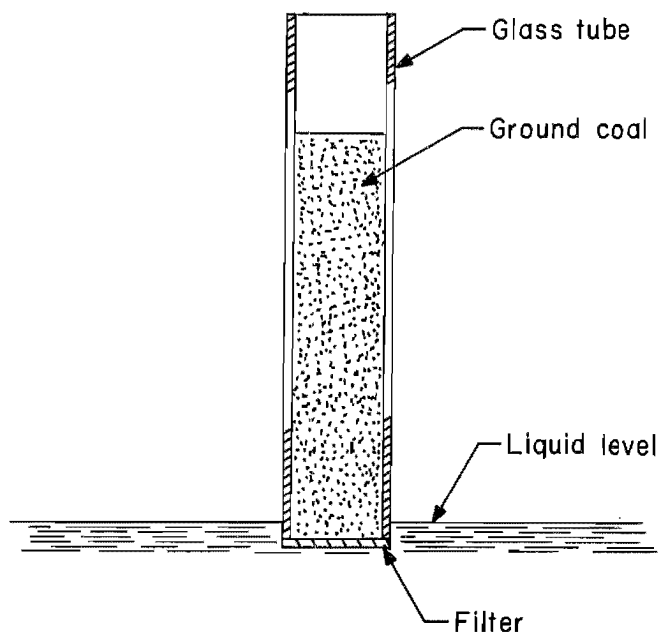


FIGURE 1. - Capillary rise test apparatus.

TABLE 1. - Precision results for the capillary rise test (Lower Kittanning coal, sample weight 2 g, surfactant 1.0 pct Surfynol 465)

Number of tests	Weight of solution absorbed, g	Gain, ¹ wt pct	Number of tests	Weight of solution absorbed, g	Gain, ¹ wt pct
1	0.27	13.5	4	0.33	16.5
3	.28	14.0	6	.34	17.0
2	.29	14.5	3	.35	17.5
3	.30	15.0	3	.36	18.0
2	.31	15.5	1	.37	18.5
2	.32	16.0	1	.39	19.5

Average weight gain: 0.33 g. Standard deviation: 0.03 g.

¹(wt gain × 100) divided by sample wt.

Repeatability of the rise test was found satisfactory. The results of 31 tests, conducted on coal from the Lower Kittanning Seam, are shown in table 1. The surfactant used was Surfynol 465⁴ diluted to 1.0 wt pct concentration in distilled water. For each 2-g coal sample, the measured weight of liquid absorbed ranged from 0.27 to 0.39 g with an average of 0.33 g and a standard deviation of 0.03 g. One source of error, a correction of 0.1 g, was required to account for the liquid retained by the filter disks. In tests involving only the tubes and attached filter, the weight of liquid retained by the filter ranged between 0.05 and 0.15 g. This 0.05-g variation is the main error in weight gain determinations.

SINK TEST

The sink test was adapted from the Draves (13) test used in the textile industry. Its use for measuring coal wettability was apparently first proposed by Walker (32). The procedure consists of depositing powdered coal on a liquid surface and timing how long it takes to sink beneath the surface. Typically, 50 to 100 mL of test liquid and 0.3 to 0.5 g of coal were used. To minimize clumping

of the coal, a flour sifter was often used to feed the coal. Additional details are given by Papic (28) and more recently by Glanville (15-16).

CONTACT ANGLE MEASUREMENT

The contact angle (fig. 2) formed between a liquid drop and a solid surface is often a measure of wettability (18, 29, 31). The angle formed ranges from 0° for complete wetting to over 90° for non-wettable surfaces. The results reported in this publication were obtained from the contractor; details on the procedures are given in the contractor's final report (14) to the Bureau.

ZETA POTENTIAL MEASUREMENT

The zeta potential, the electrical charge surrounding particulate matter in water, has been used by others to study the flocculation properties of coal suspensions (3, 35). The results in this report were obtained by Bureau contractors who used commercially available apparatus and standard techniques. Details are given in the contractors' final reports (14, 25).

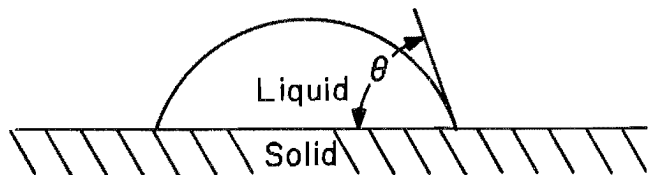


FIGURE 2. - Contact angle, θ .

⁴Reference to specific brands is made for identification only and does not imply endorsement by the Bureau of Mines. Suppliers of surfactants used in this study are listed in the appendix.

APPLICATION OF CAPILLARY RISE THEORY TO WATER-WETTABLE SUBSTANCES

Preliminary experiments were conducted on a variety of materials--quartz dust, taconite ore, oil shale, and coals from

different seams--to investigate the characteristics of the capillary rise test. An unexpected result was that a few coals

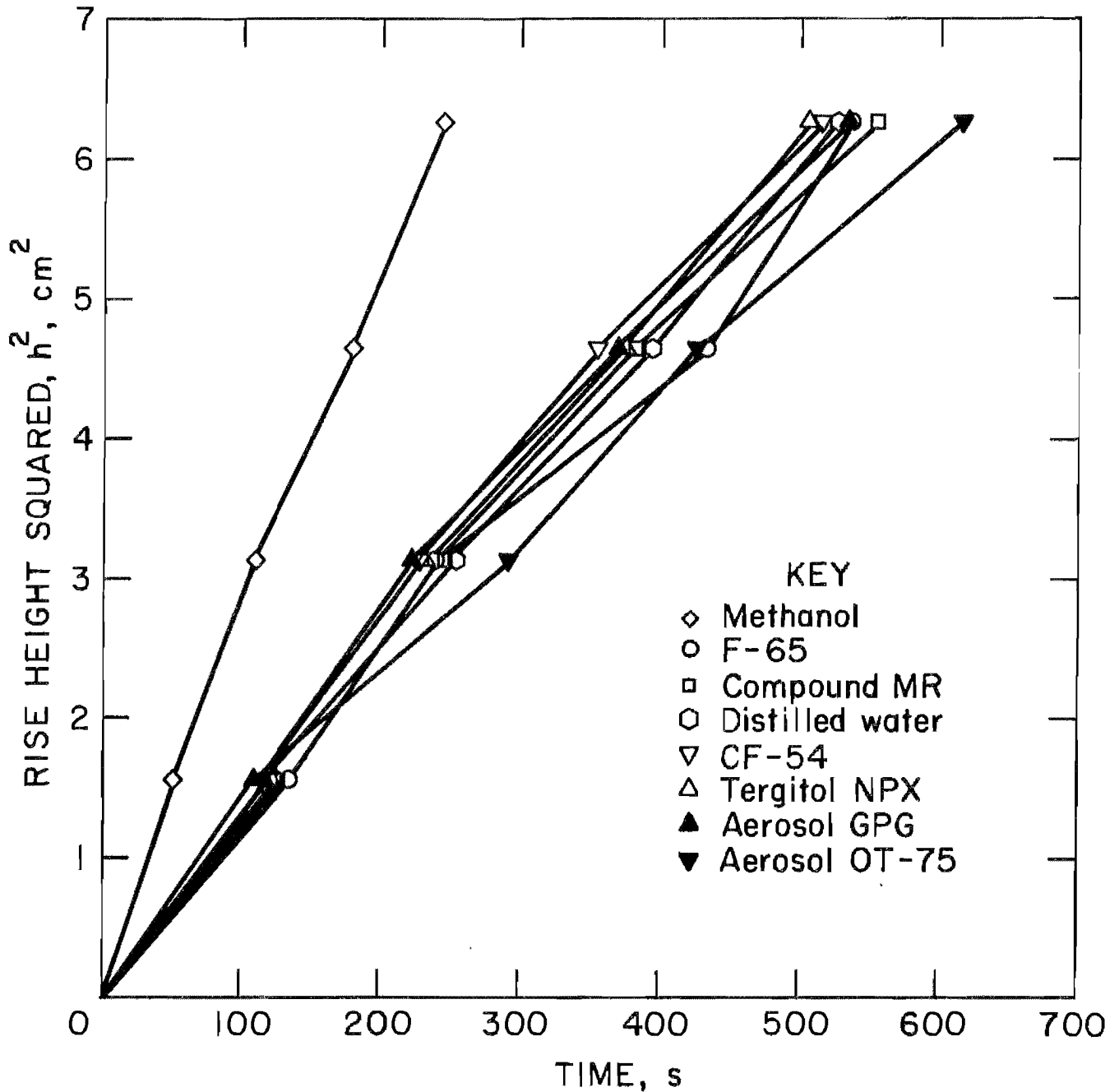


FIGURE 3. - Capillary rise test results for a wettable coal with six surfactants at a concentration of 0.10 pct.

were wet by water alone.⁵ One of the wettable coals, obtained from a seam in Indiana, was tested more extensively with methanol, water, and aqueous surfactant solutions. The time for the liquid to rise to specified column heights was determined. Results for surfactant concentrations of 0.10 and 1.0 pct are shown in figures 3 and 4, where the square of rise height, h^2 , is plotted as a function of time. The use of the square of rise

height is based on a model developed by Washburn (33), who assumed that a column of powder consisted of a bundle of capillaries, having a mean radius r , and derived the relationship

$$h^2 = \frac{r\gamma \cos\theta}{2\eta} t, \quad (1)$$

where γ is the liquid surface tension, t is time, θ is the advancing contact angle, and η is the liquid viscosity.

⁵Other tests conducted later showed that a material that appears wettable in one type of test, such as capillary rise, may appear nonwetable in another, such as a sink test. Throughout this publication wettability and nonwettability are defined in terms of the capillary rise test, unless otherwise specified, or unless a different definition is clear from the context.

As predicted by equation 1, all the plots tend to be linear. Except for Aerosol OT-75, the results from the surfactant solutions are not significantly different from those obtained using water alone. In figure 4, however, all the results at a 1.0-pct concentration are different from those with water alone. Two of the surfactants performed better than

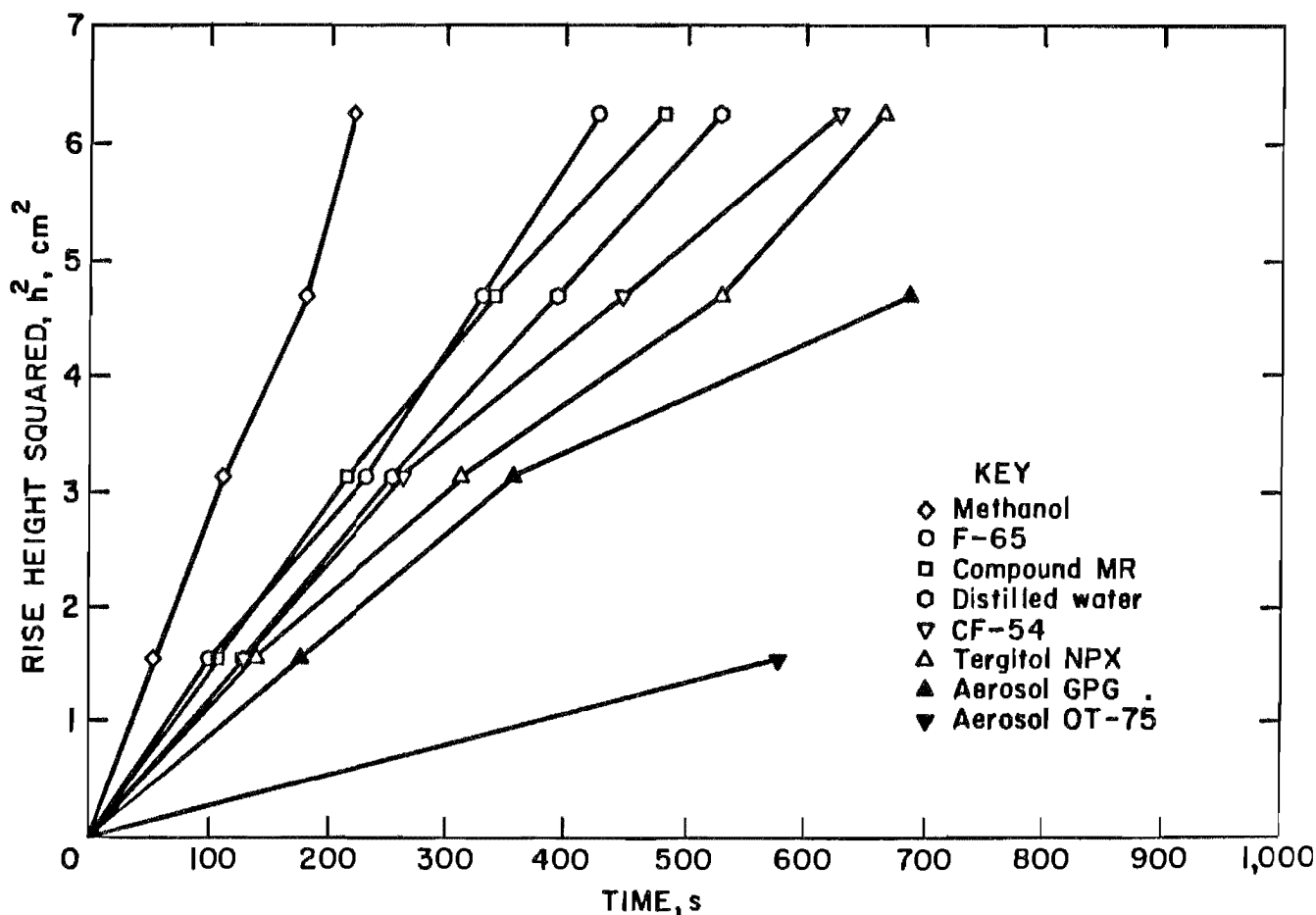


FIGURE 4. - Capillary rise test results for a wettable coal with six surfactants at a concentration of 1.0 pct.

water, three were somewhat worse, and one, Aerosol OT-75, exhibited substantially poorer performance. Note also that methanol performed much better than water or any of the surfactant solutions.

One attempt to explain these results involves the Washburn model, equation 1. Because the same coal type was used in all tests, the capillary radius, r , can be assumed constant. Therefore, the slopes of the plots should be proportional to the quantity $\frac{\gamma \cos \theta}{\eta}$, which is a velocity and was called the "penetrativity" by Washburn. Although measured values of the contact angle, θ , are not available for the particular coal, we can assign values of θ for methanol and Aerosol OT-75.

Our experience with methanol indicates that it generally wets all coals more rapidly than other liquids we have tried. For such liquids it is common practice to assume $\theta = 0^\circ$ and $\cos \theta = 1$ (9, 26). For Aerosol OT-75 Feldstein (14) measured the contact angle for 30 different coals and found that $\theta = 0^\circ$ except for one that had a contact angle of 16° ($\cos 16^\circ = 0.96$). Assuming that the contact angle is 0° and $\cos \theta = 1$ for both methanol and Aerosol OT-75, the slopes of the plots of these two liquids should be proportional to the quantity γ/η . Values of γ/η for all the test liquids are given in table 2. Note

TABLE 2. - Physical properties of test liquids

Liquid	Surface tension, γ , dyn/cm	Viscosity, η , cP	$\frac{\gamma}{\eta}$, cm/s
Methanol.....	24	0.60	40
Distilled water.....	72	.93	77
F-65.....	31	.93	33
Compound MR..	34	.92	37
CF-54.....	33	.92	36
Tergitol NPX.	34	.98	35
Aerosol GPG..	29	.96	30
Aerosol OT-75	29	.97	30

NOTE.--Surfactant concentrations were 1.0 pct, and all measurements were obtained at uncontrolled room temperatures ranging between 20° and 25° C.

that $\gamma/\eta = 40$ cm/s for methanol and $\gamma/\eta = 30$ cm/s for Aerosol OT-75, which difference is almost negligible compared with the approximate factor of 10 ratio between the slopes in figure 4.

In view of this result, it appears that Washburn's equation has only limited value as a model. Apparently, the equation is oversimplified and other phenomena are involved. For example, Van Brakel and Heertjes (31) have suggested that air entrapment, liquid evaporation, and capillary condensation may contribute to behavior differences among liquids. Also, the Washburn model assumes a pure, homogeneous liquid. As noted by Keller (22), the surfactant is adsorbed by the powdered coal, and a concentration gradient is established in the rising liquid. The surfactant diffusion rate in the rising liquid then limits the rise rate.

The results in figures 3 and 4 could also be explained if this particular coal, which is wettable with water alone, tended to become hydrophobic in the presence of some surfactants. Normally, the hydrocarbon tail group of the surfactant molecule attaches to the coal surface, while the hydrophilic polar group extends into the aqueous phase. As a result, the coal becomes less hydrophobic. If, however, the polar end should become attached to the coal surface, with the tail extended toward the aqueous phase, then the coal would appear more hydrophobic.

Zeta potential measurements show that coal in aqueous media generally has a negative charge (3, 14, 22, 25, 35). Consequently, cationic surfactants do not work well (28) because the positively charged hydrophilic polar group attaches to the negative coal surface, making the coal more hydrophobic.

Because Aerosol OT-75 is an anionic surfactant, it seems unlikely that its negative polar group would be attracted to the negative coal surface. Keller (22), however, has pointed out that coal, because of its heterogeneous composition, has a mixture of positive and negative surface sites, even though the net surface charge is negative. Keller further

describes how anionic surfactants might be adsorbed on some positive sites with their hydrophobic tails toward the aqueous phase and how this phenomenon could be used for coal dewatering.

Experiments conducted on quartz dust showed that it also could be made less wettable with increasing concentrations of Compound MR as shown in figure 5. None of the tested concentrations gave any significant wettability increase, and wetting decreased at concentrations equal to or greater than 0.1 pct; in other words, the quartz dust became

increasingly hydrophobic with increasing surfactant concentration.

These results lead to two general conclusions: First, as pointed out by Van Brakel (31), available pore space models, including the Washburn equation, are unable to provide even a satisfactory qualitative description of what is observed. Second, the use of surfactants indiscriminately is no guarantee of improvement in wetting behavior. In fact, as shown in figures 4 and 5, wettability may be inhibited for some material and surfactant combinations.

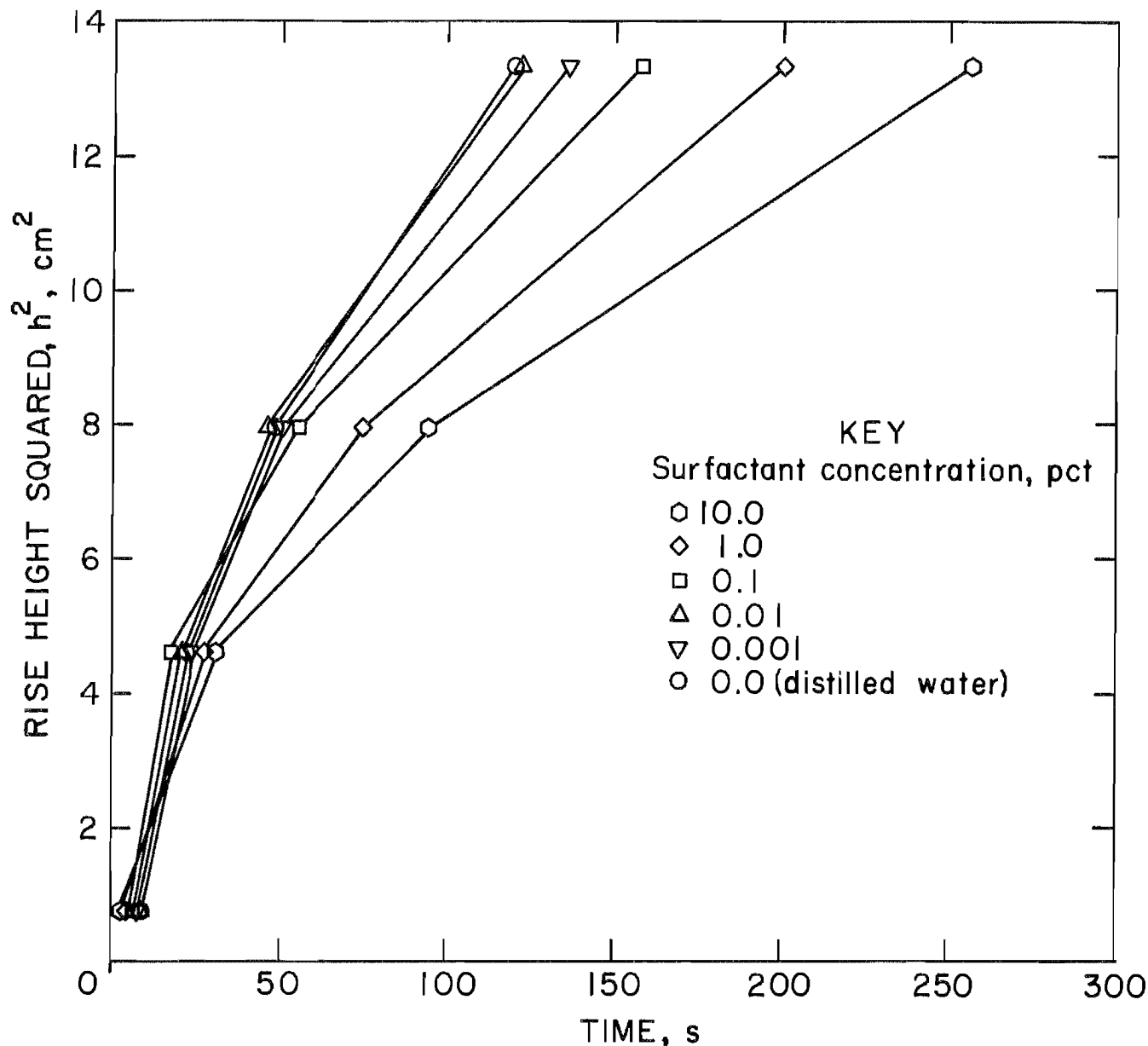


FIGURE 5. - Capillary rise test results for quartz dust with Compound MR.

SURFACTANT CONCENTRATION AND TEST DURATION EFFECTS

Following the recommendations of suppliers, many of the initial tests were conducted at surfactant concentrations of 0.1 pct or less. The results with several coals and wetting agents were disappointing in that most combinations produced insignificant wetting in short-term rise tests of 10 min or less. Additional testing then showed that with some combinations significant wetting occurred only upon increasing the surfactant concentration, the test time, or both.

SURFACTANT CONCENTRATION EFFECTS

Two difficult-to-wet coals were obtained from the Pittsburgh and Lower Kittanning Seams. Neither was wet significantly by any surfactant solution at concentrations below 0.1 wt pct. Significant weight gains in short-duration rise tests were obtained only by using concentrations greater than 0.1 pct, as shown in figure 6. Even then, surfactants such as Compound MR and DC-13 gave significant

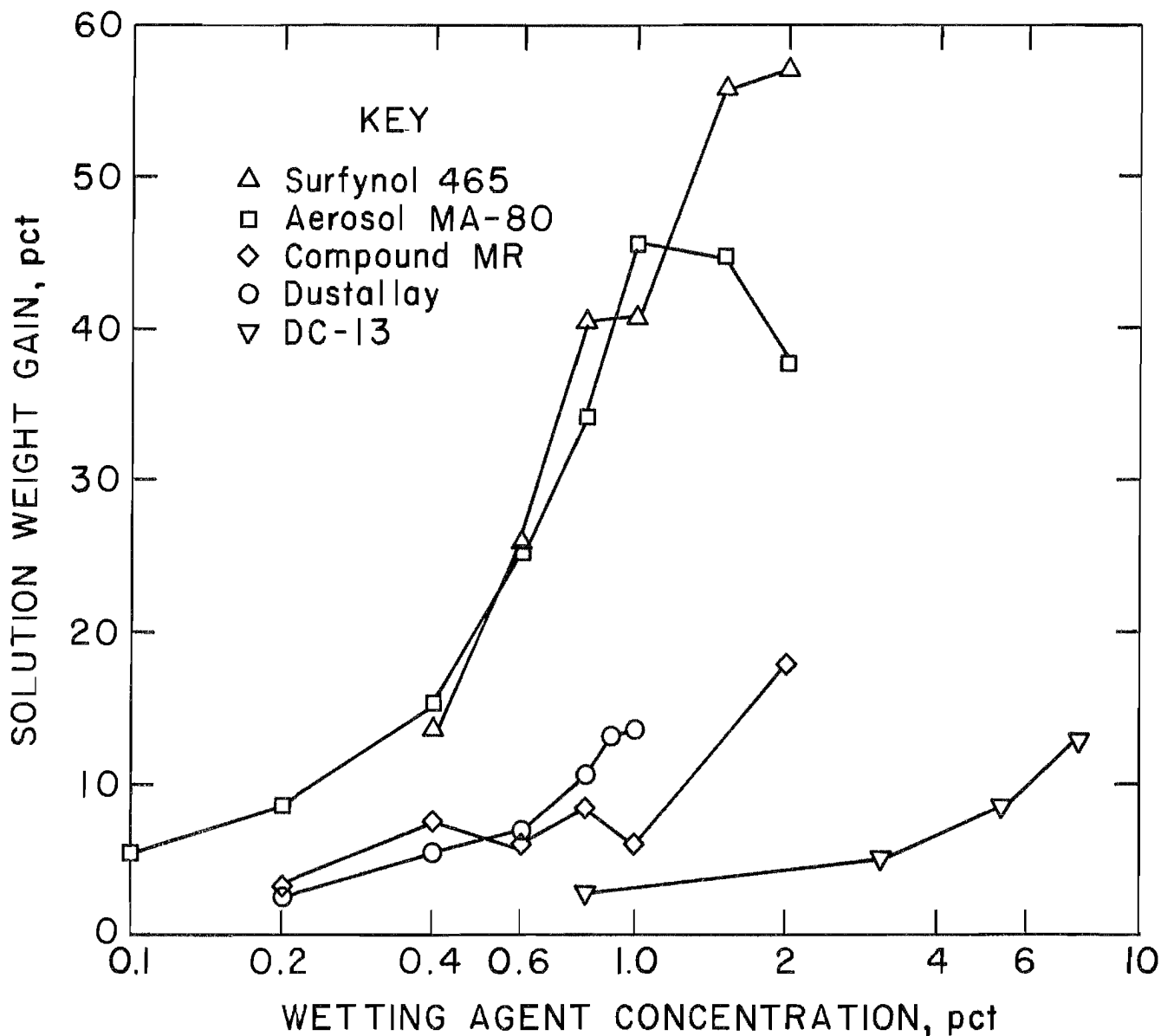


FIGURE 6. - Effect of wetting agent concentration for 10-min capillary rise period.

weight gains with Lower Kittanning Coal only at concentrations well over 1.0 pct. However, in the case of DC-13, the tested concentration of 7.7 pct represents an actual wetting agent concentration of 1.0 pct because this surfactant solution, as supplied, contains only about 13 pct active ingredients.

Although the examples shown in figure 6 exhibit some increase in wetting effectiveness with concentration, it is important not to infer from these results that other coal and surfactant combinations will always behave similarly. Kost (25) observed, for example, no significant weight gains at all for coal from the

Sewell Seam when tested with 27 different surfactants at 1.0-pct concentration.

DEPENDENCE OF CAPILLARY RISE ON TEST TIME

In some dust control applications, such as seam infusion, the rate of water absorption over long periods is of interest. Consequently, rise tests of up to 48 h were conducted to observe the behavior of various coal and surfactant combinations. Standard rise test procedures were used except that, at specified intervals, the tubes of coal were removed from the test liquids, weighed, and returned.

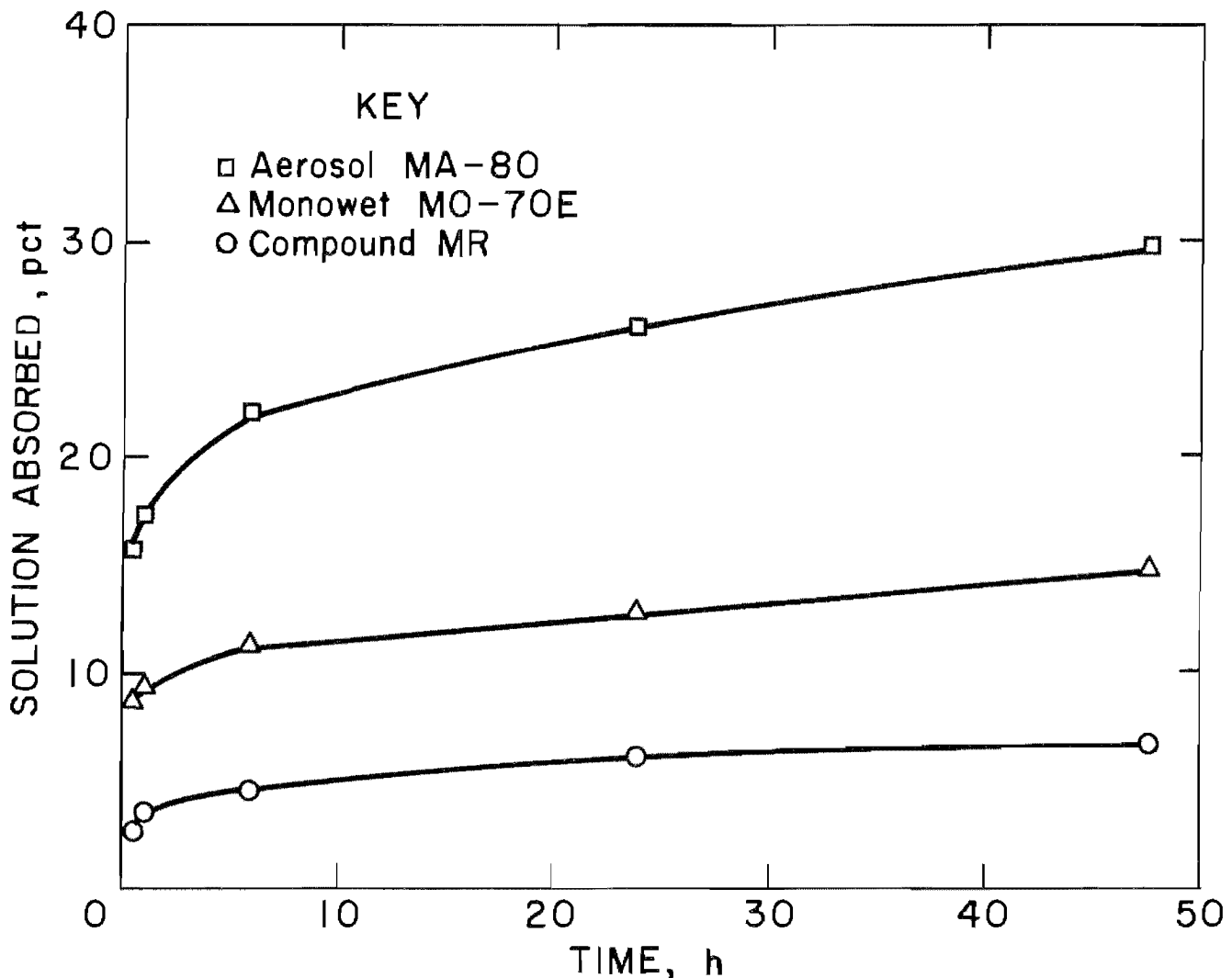


FIGURE 7. - Capillary rise test results for three surfactants and Pittsburgh Seam Coal for a 48-h test period.

The results, shown in figure 7, for three surfactants and Pittsburgh Seam Coal represent most long-term tests. Note the increasing weight gains for the entire 48 h. A theoretical limit on rise height occurs when the capillary rise force, due to surface tension, is equal to the liquid weight in the column. Weber (34) derived a relationship for this maximum height, H, as

$$H = \frac{2\gamma \cos \theta}{r\rho g}, \quad (2)$$

where γ is the surface tension, θ is the contact angle, r is the capillary radius, ρ is the liquid specific gravity, and g is the acceleration due to gravity. In general, equation 2 cannot be

evaluated for powders because neither the contact angle nor the capillary radius is measurable.

Because the maximum rise height cannot be calculated and may require impractical test time to be measured, these data verify that test periods of only a few minutes provide adequate relative measures of long-term performance.

In some situations, the effectiveness of a surfactant may depend on the rate of wetting at the moment of application. Therefore, a variation of the test was devised to observe wetting characteristics in those initial seconds and minutes. In this method an aluminum foil dish, containing the surfactant solution,

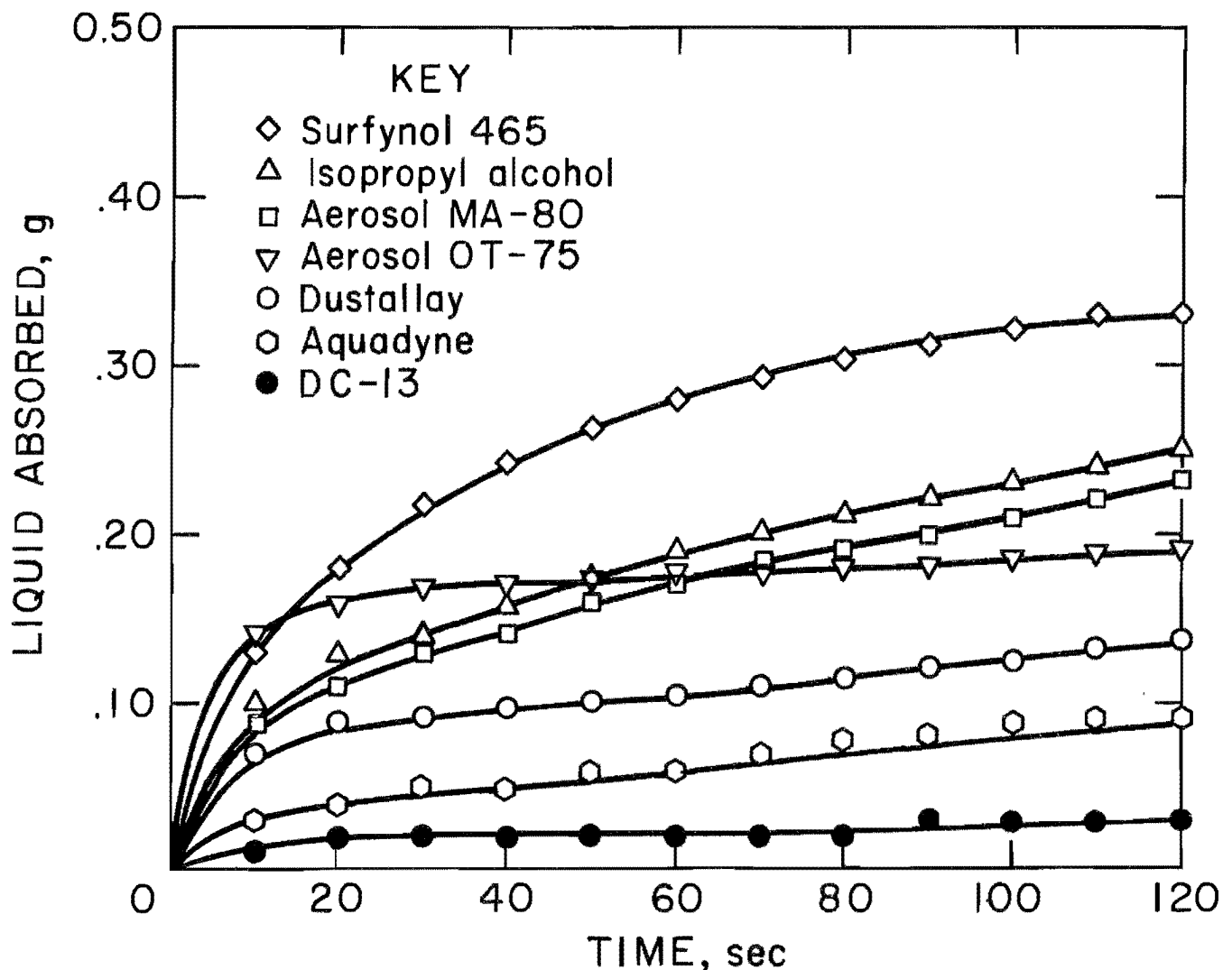


FIGURE 8. - Capillary rise test results in the initial 2 min for seven surfactants.

was placed on a balance, and its initial weight was recorded. The tube containing the coal was then dipped into the surfactant, and the change in weight of the dish was recorded at preselected intervals, usually every 10 s.

The results of one test series are shown in figure 8, where the coal's weight gain is shown as a function of time for six surfactants and isopropyl alcohol. For this coal, which was from the Lower Kittanning Seam, Surfynol 465 was the most and DC-13 the least effective.

PARTICLE SIZE EFFECTS

Coal obtained from an underground mine in Utah was milled to obtain two different size fractions: One was designated as "coarse" and the other as "fine." The size distributions of the two, determined by wet screening, are given in table 3. Note that 23 pct of the fine-grind sample was smaller than 325 mesh (44 μm), while only 4 pct of the coarse grind was smaller than that. Capillary rise tests were conducted with distilled water, which verified that this coal was not wettable.

TABLE 3. - Particle size distributions for Utah coal, weight percent

Tyler mesh size	Fine grind	Coarse grind
Plus 20.....	8	9
Minus 20 plus 35.....	11	33
Minus 35 plus 100.....	28	37
Minus 100 plus 150.....	7	6
Minus 150 plus 200.....	10	5
Minus 200 plus 270.....	6	2
Minus 270 plus 325.....	4	2
Minus 325.....	23	4
Sample loss.....	3	2
Total.....	100	100

The most interesting result is that obtained for Aerosol OT-75 in the first seconds of the test, which shows an initial high wetting rate which levels off after about 20 s. The reason for this behavior is unknown. This is an important result because initial wetting rates might be critical in applications such as continuous-mining-machine wet-head sprays. The behavior is not predictable from long-term rise tests. Because the time resolution was only on the order of 10 s, better methods are needed to pinpoint behavior differences among surfactants in the first few seconds.

The rise tests were then conducted on both grinds with solutions of 14 surfactants at weight concentrations of 0.10 pct in distilled water. The weight of surfactant solution absorbed after 48 h was determined. Also, estimations of the void volumes were obtained from rise tests with isopropyl alcohol which completely wet the coal samples.

Table 4 shows the rise heights, and weight gains for both grinds and also the coarse-to-fine weight gain ratios. Note that the rise heights tend to be unreliable indicators of solution absorbed because of irregular wetting within the column. Sometimes the outside of the coal appears wet while the column contains pockets of dry coal. The reverse also happens, with the outside dry but an inner core having absorbed some solution. As a result, weight rather than height gives more precise results.

Except for D-Dust, the coarse grind coal absorbed more of the surfactant solutions than did the fine. The coarse-to-fine weight gain ratios ranged from

TABLE 4. - Capillary rise test results for Utah coal

Liquid ¹	Coarse grind		Fine grind		Weight gain ratio, coarse/fine
	Rise height, mm	Weight gain, pct	Rise height, mm	Weight gain, pct	
Aerosol MA-80.....	25	32.5	25	18.0	1.81
Surfynol 465.....	13	21.8	6	10.3	2.12
Aerosol AY-65.....	25	20.1	25	10.3	1.95
Aerosol OT-75.....	19	17.0	9	10.1	1.68
Monowet MO-70E.....	15	15.9	8	9.4	1.69
Dustallay.....	10	15.1	10	6.9	2.19
Pluronic L-72.....	13	14.9	7	6.4	2.33
Gemtex SC-40.....	11	14.2	11	8.8	1.61
Product 55.....	15	12.3	15	6.4	1.92
Tryfac 610-K.....	5	11.8	3	6.0	1.97
Aquadyne.....	9	11.8	3	5.1	2.31
Local ²	5	10.1	5	6.4	1.58
Coal Dyne 100.....	10	9.9	10	9.0	1.10
Compound MR.....	7	8.8	5	6.8	1.29
D-Dust.....	9	6.1	4	6.4	.95
Isopropyl alcohol...	25	32.7	25	28.9	1.13

¹Surfactant concentration in distilled water was 0.1 pct.

²Locally available surfactant near mine, source unknown.

NOTE.--Average of surfactant ratios = 1.77.

0.95 to 2.33; the calculated average was 1.77. There are at least two explanations for these differences: one based on void volume, and one related to particle surface areas.

In the Washburn relationship (equation 1) because the square of the wetted column height, h^2 , is proportional to the capillary radius, r , we can infer that weight gain will increase with an increase in the capillary radius or void volume. However, the alcohol wetting data suggest that the void volume ratio is only 1.13, which is too small to account for the average wetting solution weight gain ratio of 1.77 or ratios for individual surfactants as high as 2.33.

A more likely explanation is that the fine grind coal has a greater total

surface area than the coarse grind coal. This area provides proportionately more adsorption sites for surfactant molecules. Surfactant adsorption and the resulting concentration gradient is a major limit on wetting rates and liquid absorbed. If the rise tests continued indefinitely, we would expect the maximum weight gains to be similar for both the fine and the coarse samples and consistent with the void volume and with equation 2.

Regardless of the actual explanation, the data in table 4 demonstrate that particle size is an important variable in liquid absorption and must be accounted for when comparing results between different coals or between different samples of the same coal.

MINE WATER EFFECTS

Although most tests were conducted with distilled water or distilled, deionized water, some tests were conducted with water obtained from underground mines. Generally, a sample of water used for

dust control was also obtained with coal samples. Figure 9 compares weight gains for Blue Creek Coal and three surfactants diluted with distilled and mine water.

Note that this coal was slightly wettable (5 pct absorbed) by the mine water, but not by the distilled water. At most concentrations for Surfynol 465 and Aerosol MA-80, greater weight gains were measured for the distilled water. In the case of D-Dust, small gains were measured as concentration increased in distilled water, but no significant changes were obtained with mine water at various concentrations.

The mine water's composition is unknown. This particular sample contained substances that tended to inhibit wetting agent effectiveness, but others have observed both increased and decreased effectiveness associated with specific water properties such as hardness and pH. Feldstein (14) measured selected properties of water samples from 30 different mines. He found that pH ranged between 4.8 and 8.1, specific conductance was 46 to 1,400 $\mu\text{mho/cm}$, and calcium equivalents were between 0 and 3.7 mM/L. He further measured significant variations in sink time as a function of solution pH. Others (15) have observed significant reductions in sink time when calcium ions were added to some surfactant solutions. Although Kost (25) observed some wetting improvements with added calcium, he concluded that water hardness was not a significant factor in capillary rise tests.

Because of this observed variability in wetting performance with water properties, it is apparent that surfactant selection should include checks for compatibility with the available mine water.

CORRELATION AMONG DIFFERENT WETTABILITY TESTS

Analysis of these results has revealed only minor correlations among the four different tests; in other words, these tests agree little on which wetting agents are effective for a specific type of coal. One of the few positive correlations obtained is between contact angle and column absorption, as measured by Feldstein (14) and shown in figure 10. The individual points plotted for each liquid are for different coals from an inventory of approximately 30 coals. No

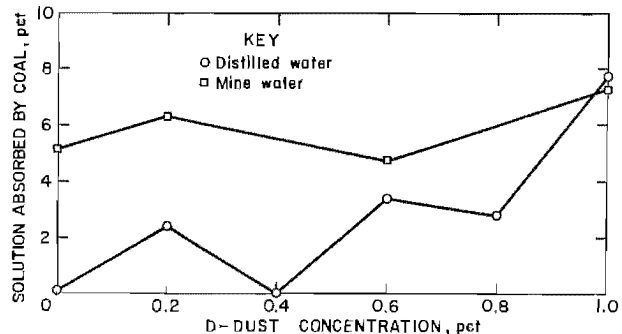
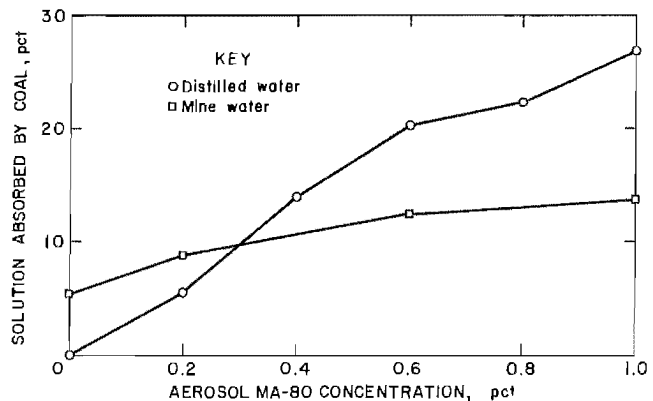
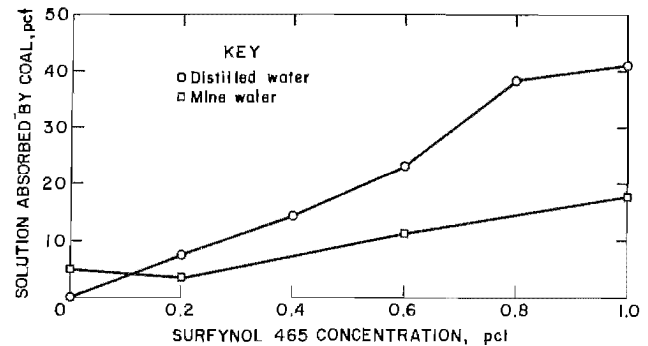


FIGURE 9. - Capillary rise test results for three surfactants mixed with distilled water and with mine water.

general, overall correlation exists between absorbed liquid and contact angle; individual plots were obtained for each liquid. Note also that the observed correlations are consistent with equation 1: Absorption increases with decreasing contact angle.

Correlation of sink test data with the results of other tests was difficult because of the incomplete sinking encountered with many coal and surfactant

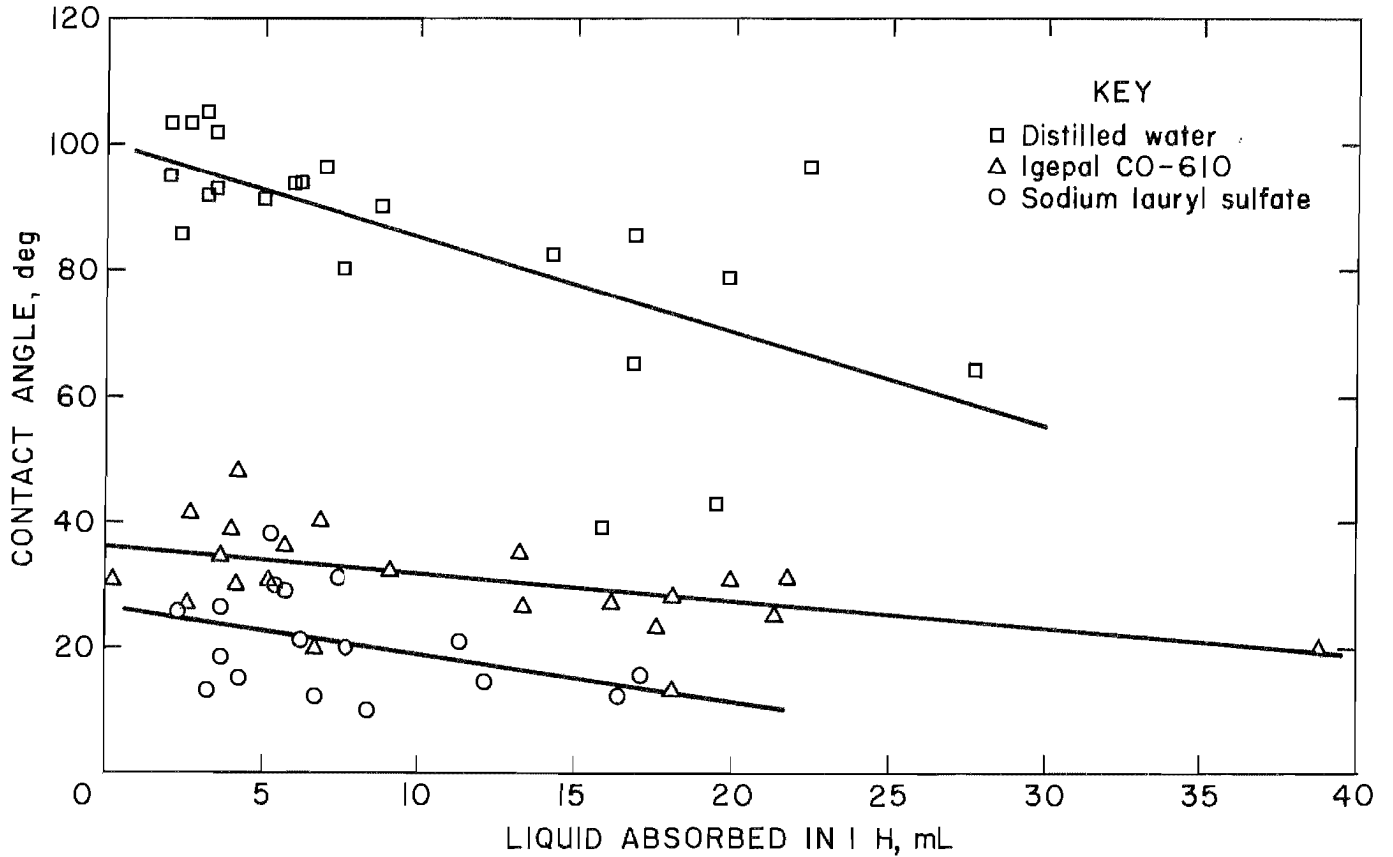


FIGURE 10, - Correlation between contact angle and column absorption measurements for various coals.

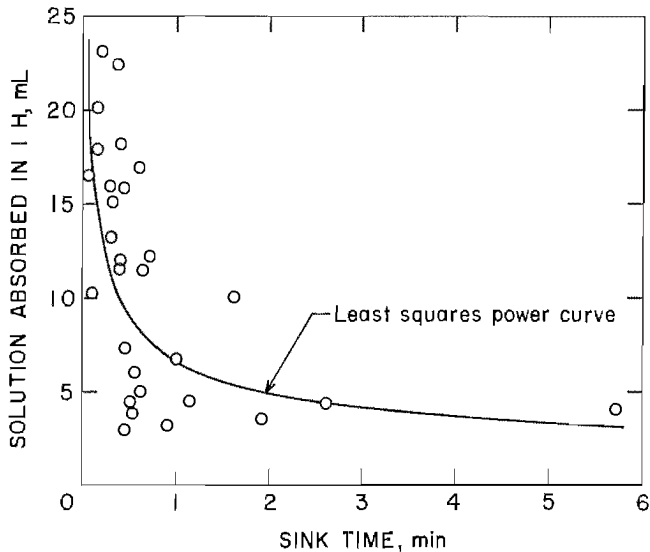


FIGURE 11, - Correlation between column absorption and sink time test results for 30 coals and Aerosol OT-75 at 0.1-pct concentration.

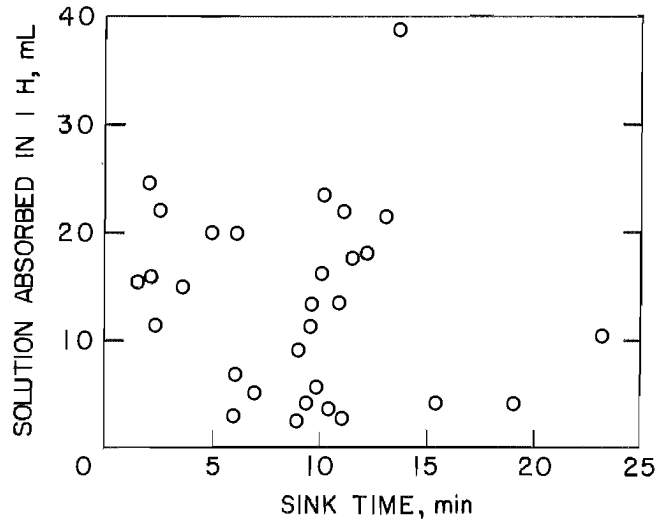


FIGURE 12, - Sink time and column absorption test results for 30 coals and Igepal CO-610 at 0.1-pct concentration.

combinations. Both Kost (25) and Feldstein (14) had this problem even with test periods of up to 2 h. Feldstein conducted sink tests on all combinations

of 30 coals and 9 surfactants. Within the 2-h time limit, only two of the surfactants resulted in sink times for all coals. The results for one of these,

Aerosol OT-75, are shown in figure 11, where a weak correlation with column absorption data is observed. The least squares power curve has a correlation coefficient of 0.62. The results for the second surfactant, Igepal CO-610 (fig. 12), show essentially no correlation with column absorption results. Attempts to fit curves for these data produce correlation coefficients of about 0.1 or less.

Of over 200 tests conducted by Feldstein (14) with the other 7 surfactants, about 65 pct involved incomplete sinking within the 2-h time limit. When

incomplete sinking occurs, a simple extrapolation cannot be carried out to estimate the time required for all the coal to sink. In practice coal sink rates tend to decrease with time--the initial rate is quite rapid; then it appears that sinking stops even though some coal may remain floating.

Because of this lack of correlation among the tests, it is concluded that each test measures specific aspects of wetting phenomena. Consequently, wettability test criteria must be selected for the intended application.

EFFECTS OF SURFACTANT MIXTURES

Experiments were conducted to determine whether or not the characteristics of individual wetting agents are retained when the agents are in a mixture. Specifically, sink and rise tests were conducted with two mixtures. Both contained Aerosol MA-80 because of its consistently good performance in rise tests with most coals; combined with the anionic Aerosol MA-80 in one mixture was a nonionic surfactant, Coal Dyne 100; in the other mixture anionic Aerosol OT-75 was used. The results for coarse and fine grinds of Utah coal (size distributions are in table 3) are presented in table 5.

In six of the eight tests, the performance of the mixture was equal to or somewhat improved over that of either surfactant alone. Two of the eight showed slightly reduced performance:

Aerosol MA-80 with Aerosol OT-75 produced a 14-pct weight gain for the fine grind coal compared with 16 pct obtained with Aerosol MA-80 alone, and with Coal Dyne 100 it produced, for the fine coal, a sink time of 24 s compared with 21 s for Coal Dyne 100 alone. These two small reductions can be attributed to experimental error and are not considered significant.

Note the large improvement for sink times obtained with the Aerosol MA-80 and Aerosol OT-75 combination. When used alone, Aerosol MA-80 produced only incomplete sinking with both coal grinds, and Aerosol OT-75 alone produced sink times of 20 and 315 s for the coarse and fine grinds, respectively. The combined surfactants produced sink times of 5 and 77 s for the two grinds. This result is

TABLE 5. - Capillary rise and sink test results for mixtures of two wetting agents

Surfactant ¹	Rise test ² weight gain, pct		Sink test ³ time, s	
	Coarse grind	Fine grind	Coarse grind	Fine grind
Aerosol MA-80.....	26	16	474	484
Coal Dyne 100.....	<5	<5	4	21
Aerosol OT-75.....	20	12	20	315
Aerosol MA-80-Coal Dyne 100..	30	17	4	24
Aerosol MA-80-Aerosol OT-75..	31	14	5	77

¹Concentration of individual surfactants in all solutions was 0.1 pct, so total surfactant concentration in mixtures was 0.2.

²Duration 24 h.

³Duration 10 min.

⁴Percent estimate of material floating after 10 min.

consistent with those of Feldstein (14), who observed large improvements when Aerosol OT-75 was combined with other surfactants.

Although the large improvement in sink times for the Aerosol MA-80 and Aerosol OT-75 combination is not explained, the small improvements in other tests are assumed to be because total surfactant in the mixtures is 0.2 pct compared

with 0.1 pct for the single-surfactant solutions.

The significance of these results is that the mixtures did well in both rise and sink tests. Accordingly, instead of requiring a single surfactant to meet specific performance criteria, two or more surfactants can be combined to meet those criteria.

DISCUSSION

Four different laboratory tests were compared for predicting surfactant wetting effectiveness. The results obtained by Bureau contractors indicate that neither contact angle nor zeta potential measurements appear to be generally useful. The problem with zeta potential measurements is their interpretation relative to coal wetting. One problem with the contact angle was the inability to make usable test specimens for all types of coal. A second problem was the difficulty of making angle measurements on porous samples. Also, both contact angle and zeta potential provide information on coal wetting similar to such limited measurements as coal moisture, ash content, solution viscosity, and surface tension. Property measurements can help explain behavior differences in coal and surfactant interactions, but no single one taken alone is sufficient for selecting wetting agents.

Because the laboratory results show no general relationship between sink time and capillary rise performance, neither appears suitable to predict surfactant performance in all dust control situations. However, these test results can

be used to choose effective surfactants for selected applications as long as the physical mechanisms and conditions match those of the test. Capillary rise test results can be used, for example, for situations involving water penetration into the coal, such as seam infusion. The sink test should be used mainly for coal penetration into water such as particle capture by spray droplets. In this regard, however, we agree with Stulov (30) that surfactants probably do not affect particle attachment and retention on the water drop surface but instead promote particle sinking into the water drop, which continually renews the liquid surface and reduces the probability that the particle will rebound from previously deposited dust. As Stulov suggested, the use of surfactants in sprays is likely to be most effective at high dust concentrations.

The sink test is limited by incomplete sinking with many coals. This phenomenon was apparently not observed and reported by others (15, 28), probably because their investigations involved only one coal or a few different coals that happened not to exhibit this problem.

CONCLUSIONS

This study emphasized the capillary rise test. Attempts to describe our results in terms of the Washburn equation (33) were not generally successful; this result agrees with the conclusions of others (31) that pore space models are inadequate. The weight of moisture absorbed was a more reliable indicator of wetting effectiveness than the height

the liquid rose in the sample columns, because of irregularities in the column. Also, factors such as time, surfactant concentration, coal particle size, and water composition were shown to affect performance.

The indiscriminate use of surfactants must be avoided. Results obtained with

the rise test show that some coals and other substances that are readily wettable by water alone become hydrophobic in the presence of some surfactants, especially at concentrations greater than 0.1 pct. This result indicates that any expected benefits from surfactants should be verified.

Wetting agents apparently retain their individual characteristics when mixed. This means that instead of requiring a single surfactant to meet specific performance criteria, surfactant mixtures can be used to meet those criteria.

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APPENDIX.--SURFACTANT SUPPLIERS

<u>Supplier</u>	<u>Surfactant</u>
Air Products and Chemicals, Inc. Box 538 Allentown, PA 18105 (215) 398-4911	Surfynol 465
American Cyanamid Co. Berdan Ave. Wayne, NJ 07470 (201) 831-1234	Aerosol GPG Aerosol AY-65 Aerosol MA-80 Aerosol OT-75
BASF Wyandotte Corp. Industrial Chemicals Corp. 1609 Biddle Ave. Wyandotte, MI 48192 (313) 282-3300	Pluronic L-72
Dowell Division of Dow Chemical Box 45828 Houston, TX 77001 (713) 433-3646	F-65
DuBois Chemicals DuBois Tower Cincinnati, OH 45202 (513) 762-6000	D Dust
Emery Industries, Inc. Chemical Specialties Group Box 628 Mauldin, SC 29662 (513) 762-6200	Tryfac 610-K
Finetex Inc. 418 Falmouth Ave. East Paterson, NJ 07407 (201) 797-4686	Gemtex SC-40
GAF Corp. Chemical Products 140 West 51st St. New York, NY 10020 (212) 582-7600	Igepal CO-610
Johnson-March Corp. 3018 Market St. Philadelphia, PA 19104 (215) 222-1411	Compound MR

<u>Supplier</u>	<u>Surfactant</u>
Mona Industries, Inc. 65 East 23d St. Paterson, NJ 07524 (201) 274-8220	Monowet MO-70E
Motomco, Inc. 267 Vreeland Ave. Paterson, NJ 07513 (201) 345-6202	Aquadyne
Preiser Scientific, Inc. Jones and Oliver Sts. St. Albans, WV 25177 (304) 727-2902	Coal Dyne 100
Rohm and Haas Co. Independence Mall West Philadelphia, PA 19105 (215) 592-3000	CF-54
Scholler Bros., Inc. Collins and Westmoreland Sts. Philadelphia, PA 19134 (215) 739-0900	Product 55
Spartan Chemical Co. 110 North Westwood Ave. Toledo, OH 43607 (419) 531-5551	DC-13
Union Carbide Corp. Chemicals and Plastics 270 Park Ave. New York, NY 10017 (212) 551-3763	Tergitol NPX
Wen-Don Corp. Box 13905 Roanoke, VA 24034 (800) 336-5713	Dustallay