

Stabilization of Loess With Aniline-Furfural

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Aniline and furfural are organic liquids that when mixed spontaneously form a resin known as aniline-furfural resin. A ratio of 2 mols of aniline to 1 mol of furfural produces the most superior resin. Soil may be stabilized by mixing with aniline, furfural, and water, followed by compaction of the mass and an adequate curing period. Different amounts of resin produce different results with any one soil.

The loess of southwestern Iowa affords an excellent opportunity for a study of the effect of amount of clay on the stabilizing efficiency of chemical treatments. The mineralogic content of the loess is very nearly the same with the exception that the amount of clay increases systematically. Several loess samples were chosen as representative of the variation in clay content and used in the aniline-furfural evaluation studies. These soils were stabilized with different percentages of aniline-furfural resin and laboratory tests were performed to determine changes in soil properties. The results of the laboratory tests were then treated as dependent variables with the clay content and the aniline-furfural content treated as independent variables. This treatment of the test results makes it possible to draw contour lines representing soil properties on a plot of aniline-furfural content vs clay content.

The aniline-furfural stabilized soils were evaluated from unconfined compressive strength determinations in a wet and in a dry state, moisture absorption, differential thermal analysis and microscopic studies, Atterberg limits, and artificial laboratory weathering. All studies seem to indicate that the excellent stability resulting from aniline-furfural resin is mainly due to the clay-organic complexes formed. These complexes appear to be the cause of the soil-resin mass becoming considerably more hydrophobic. There is also evidence of soil grains and of grain aggregates. Differential thermal analysis shows a very distinct relationship between the resin and the clay present in the soils.

● GRAVEL AND CRUSHED ROCK have been successfully used in the construction of bases and subbases for highways and airports for many years. However, many areas are now either devoid of these materials or have had their supply nearly exhausted. Use of materials from distant areas is prohibitive because of the cost of transportation. A possible solution to this problem is the utilization of local materials such as soil. Most soils are not naturally suited for base courses and therefore require stabilization.

Southwestern Iowa is an area in which that problem exists and the Iowa Engineering Experiment Station at Iowa

State College has been conducting studies of fundamental properties and stabilization methods on Iowa soils in an effort to find a solution. These studies have been directed toward efficient and economic methods of suitable base and surface course construction of secondary roads.

Early stabilization studies indicated that artificial resins were a potential means for accomplishing a practical stabilization with satisfactory results. The most effective of these artificial resins was the resin formed by the reaction of two parts of aniline to one part of furfural.

Aniline and furfural are two liquid organic chemicals that polymerize on con-

tact to form a resin known as aniline-furfural resin or more briefly as AF. The discovery of aniline was made by Unverdorben in 1826 (23) and the discovery of furfural by Dobereiner (9) in 1830. The first report of the aniline-furfural reaction is unknown but can probably be attributed to Stenhouse (22) around 1850. Aniline reacts with aldehydes to form a group of compounds that are known as Schiff bases in honor of Dr. Hugo Schiff who was professor of chemistry in Florence, Italy.

Resins have many useful applications, including the role of an impregnating agent. Impregnation may best be distinguished from coatings or the application of adhesives by the fact that the entire body of the material treated is altered in character rather than just the surface.

One of the best examples of the use of an agent of this type is in the impregnation of soft wood with methylol urea, the monomeric unit of ureaformaldehyde resins, and then carrying out the polymerization reaction in the cells of the wood. The product will not swell, shrink, or warp with humidity changes, and the grain will not rise on wetting. The material can be worked, turned, and finished to give a product superior to the hardwoods.

Aniline-furfural resin plays a role of this type in the stabilization of soil. The chemicals are mixed into the soil separately and polymerize slowly thereafter. The resin is thus formed within the soil mass.

The use of aniline-furfural resin as a soil stabilizing agent was reported by Winterkorn (24) in 1947. The resin was used in a weight ratio of 70 parts of aniline to 30 parts of furfural and was found to be most successful with medium plastic soils. Winterkorn further concluded that the resin acts both as a binder and as a waterproofing agent. The work was successfully extended in 1949 to beach sands (25).

Mainfort (16) has discussed the Civil Aeronautic Administration's investigation of chemical admixtures for soil stabilization. With the exception of port-

land cement, AF was found to be superior to all other chemicals tested.

MATERIALS

Wisconsin Loess in Southwestern Iowa

The soils employed in this investigation came from southwestern Iowa where composite Wisconsin loess forms a massive surface deposit that mantles older loesses and pre-Wisconsin glacial deposits. The loess varies in thickness, as measured on ridges and hilltops, from 60 to over 100 ft along the Missouri River bluffs to about 17 ft toward south-central Iowa.

Over one hundred samples of Wisconsin loess have been taken along the five traverses shown in Figure 1. Control samples for determining areal property variations were taken at a depth of 2 or 3 ft below the top of the C horizon. Areal and stratigraphic variations in properties were determined by comparison and correlation of results from some 25 to 30 physical and chemical tests (6).

Test data indicate that physical and chemical properties along the east valley wall of the Missouri River are remarkably uniform both areally and stratigraphically. With increasing easterly distance away from the east valley wall the test data reflect a marked increase in plasticity, shrinkage, water-holding capacity, and in-place density. These property changes appear to be chiefly due to an increase in the amount of clay in the loess (8).

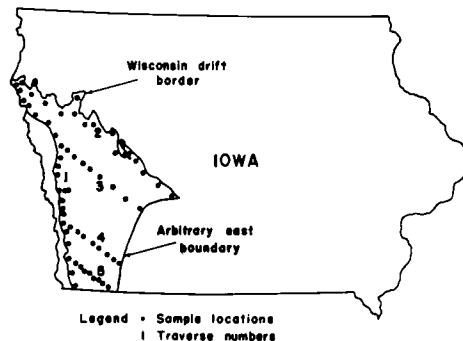


Figure 1. Locations of loess sampling traverses in southwestern Iowa.

The mineralogical nature of this Wisconsin loess seems to be quite uniform. Organic matter and soluble sulfate contents are low or non-existent. Aside from the variation in amount of clay, the principal variable in the loess appears to be carbonate content which varies both areally and stratigraphically. Carbonate contents, expressed as percent calcium carbonate by weight, were as high as 18 percent along the Missouri River to as low as 1 percent in inland samples.

The loess may be conveniently divided into two fractions of $-2\text{-}\mu$ material and $+2\text{-}\mu$ material. In general the $+2\text{-}\mu$ material contains mostly quartz, feldspar, and calcite along with a minor amount of heavy minerals. These minerals are considered to be non-reactive with large organic cations.

The $-2\text{-}\mu$ material in the loess is very similar throughout and consists mainly of clay minerals. The clay minerals in this fraction are thought to exist as a randomly interstratified mixture of montmorillonite and illite types. X-ray diffraction data indicate an increase in the montmorillonite-illite ratio with increasing clay content in the loess. Microscopic examination of the loess has shown that the clay occurs mainly as coatings on larger grains.

Cation exchange capacity is one of the most important properties of soil, a fact that has long been recognized by agricultural soil scientists (14). It can be measured quantitatively and is closely related to the physical and chemical behavior of soil. It has been related to engineering properties of Wisconsin loess (7). Southwestern Iowa loess exhibits a

range in cation exchange capacity, expressed as milli-equivalents per 100 grams of soil, from 7 along the Missouri river to 26 further inland. This increase is mainly attributed to an increase in the amount of clay in the loess.

In view of the orderly variation of the areal and stratigraphic properties of the southwestern Iowa loess and the apparent qualitative mineralogic continuity, it was decided to investigate the effectiveness of aniline-furfural stabilization on this soil by studying a few samples representing the entire area. The property study has indicated that the principal variable in the loess is the amount of clay. The soil samples were selected to provide a range in clay content from 7 to 37 percent less than $2\text{-}\mu$ material and to represent the carbonate range of 7.6 to less than 1 percent.

Several hundred pounds of each soil were obtained from the C horizon with the exception of sample 503-2 which was from the B horizon. The sample locations are described in Table 1. Samples 20-2 and 44A-1 were used in obtaining basic data necessary to further study. Sample 20-2-II is from a different stratigraphic position than 20-2 and affords a better position in the over-all representation of the range in soil properties.

The soil samples were selected from data obtained from small laboratory samples used in the initial property studies. The selections were made to present a range in $2\text{-}\mu$ clay content that increased in increments of approximately 5 percent. Subsequent analysis of representative samples taken from the larger samples used in the stabilization studies

TABLE 1
LOCATION OF LOESS SAMPLES

Sample No.	County	Township	Section	Soil Series	Thickness of Solum,* ft	Sampling Depth below Surface, ft
55-1	Harrison	Little Sioux	8	Hamburg	$\frac{1}{2}$	$2\frac{1}{2}$ -3
20-2	Harrison	St. Johns	15	Hamburg	0	60-61
20-2II	Harrison	St. Johns	15	Hamburg	0	$34\frac{1}{2}$ - $35\frac{1}{2}$
26-1	Shelby	Grove	21	Monona	2	4-5
36-1	Montgomery	Grant	14	Marshall	$3\frac{1}{2}$	$5\frac{1}{2}$ - $6\frac{1}{2}$
44A-1	Page	Morton	21	Marshall	3	4-5
46-1	Page	Amity	30	Marshall	3	5-6
503-2	Taylor	Jefferson	17	Sharpsburg	4	$1\frac{1}{2}$ -2

* Solum includes the A and B horizons where both are present.

TABLE 2
PROPERTIES OF LOESS SAMPLES

	Sample 55-1	Sample 20-2	Sample 20-2III	Sample 26-1	Sample 36-1	Sample 44A-1	Sample 46-1	Sample 503-2
Sand (2.0 to 0.074 mm), %	0.5	1.4	0.7	0.5	0.4	0.2	0.4	2.0
Silt (0.074 to 0.005 mm), %	91.1	78.8	78.9	74.8	69.6	58.9	67.9	67.9
Clay (below 0.002 mm), %	7.4	15.1	17.8	21.7	26.3	30.6	26.3	55.2
Colloidal clay (below 0.001 mm), %	6.5	14.6	17.5	21.4	25.0	29.4	24.2	37.0
Liquid limit, %	29.6	30.8	33.2	34.7	39.0	53.1	54.7	34.5
Plastic limit, %	27.3	24.6	24.0	24.0	25.6	25.7	22.0	26.7
Plasticity index, %	2.3	8.2	9.2	10.7	13.4	27.4	32.7	26.4
pH	8.0	8.7	9.2	10.7	13.4	27.4	32.7	30.3
Cat. Ex. Cap. (m.e./100 gm)	10.0	18.1	7.8	7.9	6.5	6.2	5.9	8.4
Carbonates, %	7.6	10.5	12.6	15.3	19.6	23.1	23.6	28.1
Organic matter, %	0.21	0.17	0.19	0.25	0.14	0.52	0.9	0.3
Textural classification (BPR System)*	silty loam	silty loam	silty clay loam	silty clay loam	silty clay	silty clay	silty clay	clay
Engineering classification (AASHO)	A-4(8)	A-4(8)	A-4(8)	A-6(8)	A-6(9)	A-7-6(19)	A-7-6(19)	A-7-6(19)

* Textural classification is based upon the Bureau of Public Roads System except that sand and silt are separated by the No. 200 sieve.

show the 5 percent increments to be correct. The lone exception was sample 46-1 which has a clay content very close to that of 36-1.

Table 2 lists the important physical and chemical properties of the samples. A thorough discussion of the geology, pedology, and important characteristics of the loess soils in southwestern Iowa is presented by Davidson and associates (4, 5).

METHODS OF EVALUATION

The evaluation of the ability of AF to stabilize loess was accomplished by testing 2 in. high by 2-in. diameter specimens that were molded to standard Proctor density as described below. The order of mixing chemicals and water into the soil and the method of moisture determination are discussed in the appendix.

Preparation of Mixtures

The chemicals and water were mixed into 1200 gm (oven-dry basis) of air-dry soil, producing sufficient mixture for 6 specimens. The entire quantity of furfural and about one-half of the distilled water required was added and folded into the soil with a mixing spoon. Initial mixing by hand was required to dampen the soil for the prevention of dusting. The balance of the furfural was added and mixed with the soil and furfural with a Hobart Model C-100 mixer at low speed. After mixing for 1 minute the mixer was stopped and the mixture was hand mixed for 1 minute. The aniline was then added and the mixer allowed to run for 2 minutes. Another minute of hand mixing followed and final mixing was done with another minute of machine mixing. The hand mixing was necessary because of a tendency for the mixture to adhere to the bowl and pile up.

The ratio of aniline to furfural was kept on a 2:1 molar basis because all prior (24, 25) and present research has indicated that this is the best stoichiometric relation for the formation of optimum quality AF resin. The chemicals were added volumetrically with an ac-

curacy of measurement correct to the nearest 0.1 ml. The volumes were computed on a weight basis using the specific gravity values of each chemical at 70 deg F. Desired percentages of chemical composition were produced by addition of appropriate volumes. Heights and weights of all specimens were recorded immediately after molding.

Specimen Molding and Curing

Specimens of soil-AF mixtures were molded in 2-in. diameter cylinders by means of a drop hammer apparatus described by Chu, Davidson, Goecker, and Moh (2). Experience has shown that five blows on each end of a specimen produces the most uniform density throughout the specimen. This compaction effort also produces near standard Proctor density. After compaction the molded specimen was extruded from the cylinder, weighed, and measured to the nearest 0.001 in. in height.

A curing time of 10 days was chosen on the basis of drying studies and strength studies. Figure 2 is a typical

few days of air curing and reached a constant value after 7 to 8 days.

Unconfined Compression Testing

Unconfined compression tests were made with a Riehle 60,000-lb capacity, hydraulic testing machine, and all data were read to the nearest 10 lb. The compressive load was applied at a rate of 0.1-in. deformation per minute. The maximum load causing a specimen to fail was recorded as the unconfined compressive strength.

Heights and weights of all specimens were recorded after a 10-day air curing period, and even numbered specimens were immersed in distilled water for 24 hours at room temperature. Odd numbered specimens were tested for unconfined compressive strength immediately following their physical measurements and the results reported as dry strength.

The immersed specimens were blotted dry on the surface with paper towels, measured, and weighed after the 24 hour immersion period. The specimens were then reimmersed to avoid drying during the delay period between measurement and testing. Individual specimens were removed from the immersion bath and placed in the testing machine dripping wet. The results of this test were reported as wet strength.

Atterberg Limits

Atterberg limits were determined by ASTM designations: D 423-54T and D 424-54T (1) with the exception that values are reported as percentages of the stable weight. Since the specimens to be tested were stabilized cylinders, it was necessary to break them up and grind the material with a mortar and pestle until 100 percent passed a No. 40 sieve. Mixing and kneading the material with water was very difficult but was continued until the mass was homogeneous.

Freezing and Thawing

After curing for 10 days, the specimens to be evaluated in freeze-thaw studies were immersed in distilled water

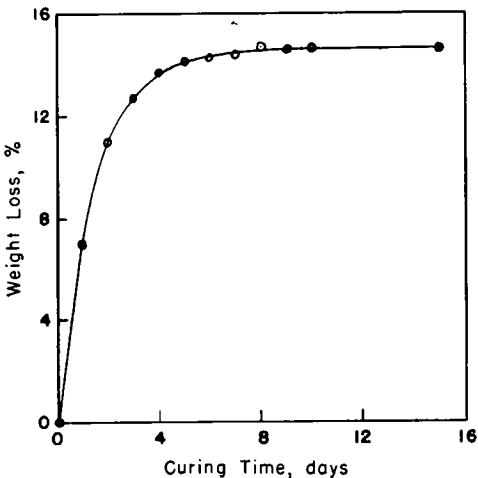


Figure 2. The total weight percent of volatile material lost in curing vs the time of curing in days for soil 20-2 using 5 percent AF.

drying curve in which drying reached a constant value after 7 or 8 days. Strength also increased rapidly during the first

for 2 hours before starting the freeze-thaw cycles. The specimens were then placed on water saturated felt pads in pans and frozen at -10 deg F for 23 hours. The pans, containing the specimens, were then removed and were partially filled with distilled water to a level just below the top of the felt pads. The specimens were allowed to thaw, exposed, at room temperature for 2 hours. The pan was then covered with Saran wrapping and kept at room temperature for another 23 hours. The foregoing 48-hr treatment was considered to be one cycle. Three specimens were tested after each cycle by weighing, measurement of height change, and immediate unconfined compression testing.

Three specimens of each soil and treatment were allowed to air dry for 30 days after nine cycles had been completed. They were then subjected to a final cycle of freezing and thawing and tested by the aforementioned procedure.

Wetting and Drying

A complete cycle of wetting and drying consisted of 24-hr air drying followed by 24-hr immersion in distilled water. The three specimens to be tested after each cycle were blotted dry on the surface with paper towels and their heights measured. They were then placed in open, individual moisture cans, and were weighed and tested for unconfined compressive strength while resting in the cans (the use of cans while testing insures the confinement of all flakes or chips resulting from testing). The moisture can containing the entire sample was placed in an oven at 100-110 deg C for 24 hours, cooled for one hour in a dessicator, and weighed. This procedure provides an accurate average moisture content determination since water does not permeate a specimen completely.

Three specimens of each soil and treatment were allowed to air dry for 30 days after nine cycles had been completed. They were subjected to a final cycle of wetting and drying and then tested.

Differential Thermal Analysis

Differential thermal analysis is generally used as an aid in the identification of clays, minerals, and other crystalline materials. The unknown sample is heated simultaneously with an inert material such as calcined alumina. Both sample and inert material are heated at a constant rate and their temperature difference is recorded on a strip chart by means of a differential thermocouple. The temperature of the sample block is recorded on a separate strip chart.

Each mineral or chemical undergoes characteristic reactions that occur at specific temperatures. These reactions are either endothermal or exothermal in nature and are revealed together with the temperature at which they occur when the two strip charts are correlated. The theory of differential thermal analysis is further discussed by Kerr, Kulp, and Hamilton (15).

Differential thermal curves were obtained from a number of AF stabilized specimens. The samples varied in amount of clay contained as well as in the amount of AF content. These samples were material salvaged from unconfined compression tests. The coarse fragments were gently ground to pass a No. 40 sieve before placing directly into the differential thermal apparatus. Curves were also obtained from a non-stabilized sample of each soil.

RESULTS

Standard methods for the evaluation of the effectiveness of soil stabilizing chemicals have not been established and correlated with field performance. Since such criteria are lacking, it is not possible to make any specific predictions concerning the field use of aniline-furfural stabilized soils. Laboratory investigations, however, do produce mixture designs that appear to give the most efficient performance for any one soil.

Contour Graphs

Exploratory work led to the conclusion

that many properties of stabilized loess depend on the amount of AF introduced and on the amount of clay contained by the native soil. It was thought that properties of stabilized loess could be related to these two variables by three dimensional diagrams, using the z-axis for the dependent variable or property to be studied, the y-axis for the AF content and the x-axis for the less than 2- μ clay content. (Hereafter clay content will be defined as all material smaller than 2 μ equivalent spherical diameter as determined by the hydrometer technique.) Three dimensional graphs are difficult to present clearly by planar diagrams. However, the use of contour lines such as are used in topographical mapping makes this possible. The AF content was plotted as the ordinate and the clay content as the abscissa. For any set of coordinates stabilized loess has a definite numerical value for each property. These values were written over their coordinates and all equal values connected, thus creating isoproperty lines.

Density

The dry density of AF stabilized loess may be expressed either as the weight of oven-dry soil particles per unit volume of stabilized soil, or as the weight of the oven-dry soil particles plus AF per unit volume of stabilized soil. In this paper the former is referred to as the dry density the latter as the stable density.

Dry density contours for aniline-furfural stabilized loess are shown in Figure 3. A three dimensional figure of this data would show a surface similar to a sugar loaf hill. By holding the AF variable constant and making a plot of density vs clay content, the curves show peaks near 22 percent clay. Apparently this effect is due to the gradation of the loess samples. Since about 98 percent of loess is silt and clay-size material, the amount of silt in a sample is closely equal to 100 minus the clay content.

The AF variable also has a pronounced effect on the dry density. If the clay variable is held constant (Figure 3), the dry density drops slowly as AF is added.

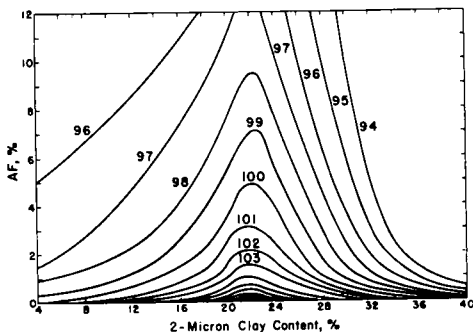


Figure 3. Dry density contours showing the relationship of dry density to AF content and clay content in loess. The values of the contours are in lb per cu ft.

Plotting the volume of soil displaced against the volume of AF added shows that AF displaces some of the soil. This plot is not linear because some of the AF fills the void spaces rather than displacing soil. The reduction in dry density is partially due to aggregation caused by the association of large organic cations, developed in the resinification reaction, and the clay minerals.

A contour plot of stable density is shown in Figure 4. The influence of gradation is much the same, but the influence of AF content on the stable density is very different from its influence on dry density. Instead of causing the stable density to steadily decrease for any given clay content, the AF causes the stable density to decrease at first and then to increase. Evidently the weight

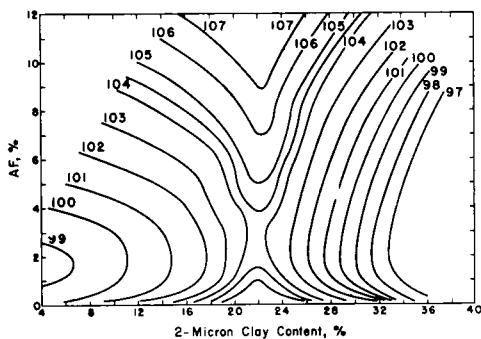


Figure 4. Stable density contours showing the relationship of stable density to AF content and clay content in loess. The values of the contours are in lb per cu ft.

loss due to displacement of soil by AF is more than overcome by the added weight due to the AF contained in the voids. The net effect of the two variables, AF and clay, is the formation of a ridge near 22 percent clay due to gradation effects, the formation of a narrow sloping trough to the left of the ridge, and a wide sloping trough to the right of the ridge. A saddle point at a clay content of 22 percent and an AF content of 2.5 percent results at the concurrence of the ridge and the sloping troughs.

Optimum Moisture

Optimum moisture content contours for standard Proctor density are shown in Figure 5. A comparison of this figure and Figure 4 shows a striking resemblance between their contour lines. Closer examination reveals that the surfaces formed are the exact opposite of each other. Figure 5 shows a valley along the 22 percent clay line and a ridge closely following the 2 percent AF line. The liquid chemicals appear to replace some of the moisture required for compaction so that less moisture is needed to cause the soil to flow for proper compaction. In all cases the total liquid added is greater than the amount of water required for natural soil.

Compressive Strength

Specimens tested for unconfined com-

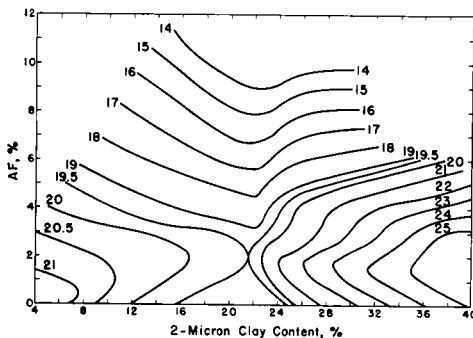


Figure 5. Optimum moisture contours showing the relationship of optimum moisture to AF content and clay content in loess. The values of the contours are in percent of the dry weight of soil in the stabilized specimens.

pressive strength after air curing were always higher in strength than those of equal AF treatment and equal clay content that were tested after 24 hours immersion in distilled water. Contour lines for immersed strength of AF stabilized loess are shown in Figure 6. The surface presented by these contours is again best described as a sugar loaf hill remarkably symmetrical to an axis along the 21 percent clay line. Gradation is shown to have an important influence on the property being studied. The influence of gradation in this case is probably an indirect effect acting through the medium of density. Density is recognized by most workers in soil stabilization as having a profound influence on strength. An examination of the surface shows that the rate of strength increases with increased AF content is much lower for low and high percentages of clay, than it is in the mid-range of clay. This also is probably primarily due to the influence of gradation on density.

Moisture Absorption

Moisture absorption after 24 hours immersion in distilled water is shown by Figure 7. Clay content is seen to have very little influence on moisture absorption until more than 23 percent clay is reached. Past this point the AF content does not exert much influence. Below 23 percent clay the AF influence is more in evidence. Below 18 percent clay the in-

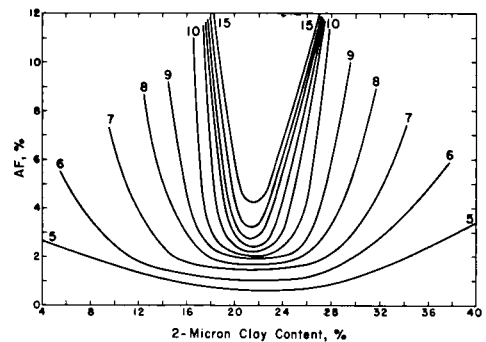


Figure 6. Immersed strength contours showing the relationship of immersed strength to AF content and clay content in loess. The values of the contours are in hundreds of pounds.

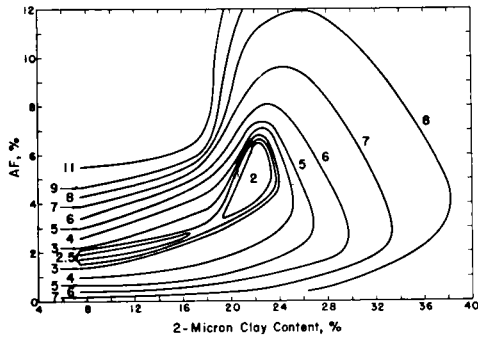


Figure 7. Moisture absorption contours showing the relationship of moisture absorption to AF content and clay content in loess. The values of the contours are in percent of the weight of the soil plus the weight of the AF.

fluence of AF is almost the same for all clay contents, in that a minimum moisture absorption occurs with a 2 to 3 percent AF treatment. In the 18 to 23 percent clay range a treatment of 1.5 to 7 percent AF appears to be quite satisfactory. The small effect of the amount of AF on moisture absorption in the higher clay ranges is most likely due to the dif-

ficulty of mixing, with a consequent poor intimacy of contact between the clay and the AF. The higher clay content loess samples showed a definite tendency to aggregate, contributing to the difficulty of mixing.

Atterberg Limits

The effect of AF treatment on the Atterberg limits is shown in Table 3. The data show that a small amount of AF greatly reduces the plasticity index in all cases. Additional AF further reduces the plasticity index until the material finally becomes non-plastic. The Atterberg limits are a basis for comparison of soil stabilizers as to their relative water-proofing ability. During the preparation of the AF stabilized specimens for the Atterberg determinations, considerable difficulty was experienced in obtaining a good mixture of water and stabilized material in all cases. As much as 30 minutes of kneading with a spatula was required.

TABLE 3
ATTERBERG LIMITS OF STABILIZED AND NON-STABILIZED SOILS

Sample No.	Aniline-Furfural Content %	Liquid Limit %	Plastic Limit %	Plasticity Index %
55-1	0	29.6	27.3	2.3
	2	NP*	—	NP
	5	NP	—	NP
20-2II	0	33.2	24.0	9.2
	1	27.6	24.0	3.6
	2	25.0	22.4	2.6
	3	26.3	24.3	2.0
	5	NP	—	NP
	7	NP	—	NP
26-1	0	34.7	24.0	10.5
	2	26.6	25.1	1.5
	5	24.8	21.0	3.8
36-1	0	39.0	25.6	13.4
	1	32.0	23.0	9.0
	2	30.9	24.2	6.7
	3	29.7	24.8	4.9
	5	NP	—	NP
	7	NP	—	NP
46-1	0	54.7	22.0	32.7
	2	33.8	26.4	7.4
	5	29.8	25.1	4.7
503-2	0	56.7	26.4	30.3
	2	37.7	29.5	8.2
	5	31.9	27.9	4.0

* Non-plastic.

Freezing and Thawing

Samples were subjected to freezing and thawing cycles and three samples were tested after 1, 2, 3, 4, 6, and 9 cycles. Three other samples were set aside after the 9th cycle and allowed to air dry for 30 days before being subjected to a 10th cycle (Table 4). The strength tabulated as zero cycles is the air dry strength of the soil.

The moisture absorbed was calculated as the gain in weight above the air dry weight of the sample and was based on the stable weight. The small amount of moisture absorption is thought to account for the remarkable durability of the specimens. There was no spalling or noticeable sample disintegration. Some samples became slightly soft where they were in contact with the moist felt pads and suffered mechanical abrasion if they were handled roughly.

The strengths of the specimens decrease considerably with the first cycle but appear to trend toward a constant value thereafter. The 30-day period of drying after the 9th cycle caused the strength following the 10th cycle to revert to a value near that found after the first cycle.

Wetting and Drying

The specimens employed in this study were subjected to an identical number of wet and dry cycles as were used in the freeze-thaw study (Table 5).

The strength, like that of the freeze-thaw specimens, drops considerably with the first cycle and remains relatively constant thereafter. After a 30-day drying period and a 10th cycle of wetting and drying, the specimens did not show any significant changes over the 9th cycle. Apparently some of the resin, products of the reaction, or unreacted chemicals are leached from the specimens during the periods of immersion. A distinct amber color was noticed in the immersion water with the depth of coloring increasing with each cycle. The discoloration is most probably due to the unreacted chemicals because resins are usually insoluble in water. An odor typical of aniline-furfural also emanated from the immersion water. Some wet-dry specimens exhibited minor shrinkage cracks but these cracks did not appear to be serious and no critical effects could be assigned to them.

The number of cycles used in both of the foregoing durability studies are con-

TABLE 4
DATA OBTAINED FROM FREEZING AND THAWING TESTS

Soil and Amount of Resin Treatment		Cycle No.	Moisture Absorbed %	Height Increase, in. x 1000	Strength, lb		
20-2II	5%	0			2800		
		1	1.4	4	1320		
		2	1.7	1	1450		
		3	1.8	4	1210		
		4	1.7	4	1030		
		6	2.5	5	1080		
		9	2.6	4	970		
		10*	0.8	0	1360		
		20-2III	2%	0			2090
				1	2.0	1	490
2	2.3			0	500		
3	2.8			4	390		
4	2.9			6	390		
6	3.1			5	420		
9	3.7			8	290		
10*	1.4			2	460		
36-1	5%			0			3380
				1	1.8	4	1930
		2	2.3	3	1730		
		3	2.6	9	1460		
		4	2.9	10	1370		
		6	3.9	13	1080		
		9	4.3	16	970		
		10*	1.6	7	1770		

* 30 days of air-drying between cycles 9 and 10.

sidered to be very severe weathering tests for 2-in. by 2-in. specimens. Winterkorn (26) has reported that four cycles with 2- x 2-in. specimens is about equivalent to 12 cycles with the 4 x 4½ standard Proctor specimens used in freeze-thaw and wet-dry tests for evaluating soil cement. The durability of the AF treated loess after nine cycles in both tests is therefore quite outstanding.

Differential Thermal Analysis

Sheeler, Handy, and Davidson (21) have reported the results of differential thermal analysis of AF stabilized loess. Briefly, it was concluded from this analysis that the aniline-furfural resin and the clay are very intimately associated and some of the resin is formed on and within the clay mineral lattice with the remainder filling the voids and cementing the loess particles together.

Microscopic Examination

Examination of stabilized specimens under a low power binocular microscope revealed some interesting phenomena. The soil particles appear to be well ag-

gregated with the degree of aggregation and size of aggregates increasing with clay content. The aggregates also appear to be surrounded and interlaced with resin.

The homogeneity of the over-all soil-resin mass is excellent in the lower clay ranges. Higher clay content soils contain some aggregates that are surrounded by resin but are devoid of resin in their centers. Fracture planes that were examined revealed that both the bonding resin between aggregates and the aggregates themselves failed in shear. These planes appear to be like a cobble-street when viewed under a microscope.

Economics

Availability and cost are two of the most important factors in any contemplated use of chemical materials. Since the use of aniline and furfural in soil stabilization would involve considerable quantities of both chemicals, the question of availability arises. A preliminary report of the Tariff Commission shows an aniline production of 132 million lb in 1955 (19). Present day production of furfural is about 50 million lb per year.

TABLE 5
DATA OBTAINED FROM WETTING AND DRYING TESTS

Soil and Amount of Resin Treatment	Cycle No.	Moisture Absorbed %	Height Increase, in. x 1000	Strength, lb		
20-2II	5%	0		2600		
		1	3.7	860		
		2	4.2	730		
		3	5.1	750		
		4	3.7	780		
		6	4.1	760		
		9	3.8	770		
		10*	4.0	740		
		20-2III	2%	0		2090
				1	3.9	395
2	4.0			330		
3	4.9			310		
4	4.5			290		
6	5.0			280		
9	4.6			320		
10*	4.9			260		
36-1	5%			0		3380
				1	2.8	1250
		2	5.7	870		
		3	7.6	650		
		4	6.7	700		
		6	7.8	570		
		9	8.1	500		
		10*	7.4	590		

* 30 days of air-drying between cycles 9 and 10.

TABLE 6
ECONOMIC COMPARISONS OF VARIOUS STABILIZING TREATMENTS FOR SOIL 20-2

Stabilizing Material	%	Treatment Cost, cents per lb	Strength after 24-hr Immersion lb	Stabilization cost, cents per 100 lb of soil
Aniline-furfural	1	19	530	19
	2	19	810	38
	3	19	980	57
Cutback asphalt	10	2.41	220	24
Portland cement	15	1.111	1780	16.7
Lime-Fly ash (1:2)	18	0.58	485	10.5
Hydrated lime	6	1.125	250	6.7
Arquad 2S	0.16	43	310	6.7
Crude amine	0.30	18	260	5.3
Armeen residue	0.38	9	330	3.5
Arquad 2HT	0.08	36	330	2.7

Aniline and furfural in car lots were priced at \$0.22 and \$0.13 per lb respectively in March of 1956 (19). This places the cost of a 2:1 resin at \$0.19 per lb. Table 6 has been prepared from data supplied by personnel of the Iowa Engineering Experiment Station who have been working with the different chemicals listed. Stabilization of loess with AF is one of the most expensive treatments listed. However, the benefits derived from the use of AF could justify the extra cost in many cases. It is conceivable that AF could be used in conjunction with a cheaper method of stabilization in building roads, the more economical method making up the majority of the stabilized material and the AF being used in areas of critical moisture situations.

Lower grades of either aniline or furfural are not available because of the nature of the processes by which they are made. Manufacturers find that they are able to sell the purest products cheaper than they can sell the chemicals contaminated with water because of the cost in transporting inert water.

Furfural plants are located in Memphis, Tenn., Omaha, Neb., and Cedar Rapids, Iowa.

Toxicity of Aniline and Furfural

The toxicity of aniline has been studied in considerable detail because of its industrial importance and uses. According to Osol and Farrar (20), the symptoms produced by the inhalation or ingestion of aniline are headache, nausea, prostra-

tion, giddiness, mental confusion, and violent neuralgic pains. If the dose has been large enough these symptoms are followed by cyanosis, excessive perspiration, loss of reflexes and voluntary movement, hurried weak pulse, rapid or irregular respiration, hemoglobinuria, and coma with dilation of the pupils. Sufficiently long survival of a large dose of poisoning produces jaundice with a great increase in the biliary pigment of the skin.

Cases of chronic poisoning develop anemia, skin eruptions, nervous symptoms, and blindness. Chronic cases lasting a number of years occasionally develop bladder cancers as a result of continuous irritation of that organ. Aniline workers exhibit a high incidence of skin tumor and cancer of the bladder. Development may come several years after exposure.

Osol and Farrar state that ingestion of as little as 0.25 cc of aniline may produce symptoms of poisoning. Recovery has followed ingestion of 75 cc, and, on the other hand, ingestion of 25 cc has proven fatal. The maximum allowable vapor concentration is 5 parts per million (17).

Furfural has very mild toxic effects compared to those of aniline. Fairhall's Industrial Toxicology (10) states that 280 parts per million of furfural causes only slight irritation to the mucous membranes of lower animals. Furfural is less apt to produce contaminated atmospheres than aniline because a higher concentration is required for toxic effects. Con-

tinued exposure to furfural can produce chronic effects, so unnecessary exposure should be avoided.

The Merck Index (17) gives the toxicity of furfural as about one-third that of formaldehyde. Furfural causes lacrimation, inflammation of the eyes, irritation in the throat, and headache. Symptoms of chronic poisoning are nervous disturbances, photosensitivity, and disturbance of vision.

First Aid and Treatment of Aniline Poisoning

Goodman and Gilman (11) state that if aniline vapors are inhaled the victim should be removed immediately to fresh air. Aniline spilled on the skin should be washed off immediately because of the danger of absorption into the body. Large amounts of water may be used, but alcohol or ether is preferable to water since aniline is very soluble in these solvents. Treatment is the same as for acetanilid poisoning. Special emphasis should be placed on shock therapy, cardiovascular system support and correction of the acute anemia with oxygen inhalation, methylene blue or ascorbic acid administration, and blood transfusion if necessary.

SUMMARY

The mechanism of aniline-furfural resin formation within the loess may be explained theoretically by assuming that the phenomena of absorption and adsorption of the chemicals by the predominant montmorillonitic type clay minerals is of primary significance. Absorbed chemicals form part of the resin within the expanding lattice of the clay minerals, and the rest of the final resinous products are formed by a reaction between absorbed and free chemicals. The formation of resin from absorbed chemicals produces a link through the crystal lattice of the clay minerals. The resin formed by the reaction between adsorbed and free chemicals produces a covering over the clay and a cementation between the clay and

surrounding particles with an aggregating effect.

The over-all result of this type of mechanism is the formation of several types of bonds in addition to those that exist in the natural soil. The natural bonds in loess are primarily those of clay to silt and clay to clay, as several investigators have found the clay to exist mainly as a covering on the larger silt particles. The bonds resulting from the resinification reaction are those of resin to clay, resin to silt, resin to large organic cations adsorbed on the clay, and resin to resin.

Evidence that this theory is correct is pointed out in the following discussion of the chemicals involved, the soil, the laboratory data, and phenomena observed by other authors.

The reaction between aniline and furfural produces large organic cations as discussed by Gieseking (12). He also states that a 2 percent treatment of soils should be sufficient to saturate all soils except those of the very highest cation exchange capacities. Large organic cations of aniline-furfural held in the exchange positions should be able to enter into polymerization reactions by virtue of their unsaturated double bonds and be linked to other aniline-furfural cations held in other exchange positions. This would create a bond between clay minerals and aggregates of soil particles.

Jordan (13) has observed that furfural is more highly absorbed by clay minerals than aniline. Furfural is also more soluble in water. The mixing study (see Appendix) has revealed that the introduction of water and furfural before aniline produces the most satisfactory results. More resin is probably formed between the unit layers of the clay minerals when this mixing procedure is used. Since furfural is more soluble in water than aniline, the introduction of furfural first should also tend to provide the most uniform distribution and absorption of chemicals.

The low shrinkage during drying and the low amount of swelling on immersion may be explained by this theory. The formation of a resin between the unit

layers of the clay minerals tends to hold the layers apart in an expanded position even after moisture removal, accounting for the low amount of shrinkage exhibited. Conversely, the re-entry of moisture provides little or no expansion, since the clay minerals are already in an expanded state.

The low moisture absorption and hydrophobic character of the aniline-furfural stabilized soils are also consistent with the theory. Saturation of the cation exchange sites with large organic cations to produce hydrophobic qualities has been discussed by Davidson (3) and Gieseking (12). The aniline-furfural reaction products include such cations, and the hydrophobic character of loess stabilized with aniline-furfural is well illustrated by the Atterberg limits and weathering tests. Moisture does find its way into the clay minerals but in small quantities only. The volume of available space is a large contributing factor to the magnitude of water absorption. Any resin formed between the unit layers of the clay minerals and in the void spaces of the soil mass prevents moisture from occupying these positions; therefore, the amount of moisture absorption is reduced below that possible before stabilization.

The theory of the formation of resins between the unit layers of the clay and on the clay is supported by the differential thermal analysis, which indicates that the resin is formed in close association with the clay. The steady gain of immersed and dry strength with increased AF content supports the contention that an enclosure and cementing of soil grains and aggregates occurs. Increased resin content tends to increase the thickness of the enclosing and cementing wall, thus increasing the over-all strength of the stabilized soil.

According to this theory stability should increase with an increase in clay content. However, the data indicate that the optimum stability occurs in the mid-range of clay content. There is an indication that aggregation of treated soil also increases with clay content. In soils having clay contents above that which

produces maximum stability the aggregation appears to be increased so that total or near total saturation of the clay minerals with the resin is prevented. This tends to reduce the effectiveness of the over-all stabilization of a soil with a high clay content by leaving some soil in the aggregates untreated, as was found to be true by visual inspection of fracture planes. Possibly, the use of a small amount of sodium ion in the mixing water would aid this situation by dispersing the clay particles so that contact between the resin and more clay would be possible.

The contour curves (Figures 3, 4, 5, 6 and 7) should aid in the design for any contemplated use of aniline-furfural. The use of these curves requires a knowledge of the clay content of the soil only. Primary consideration should be given to strength and moisture absorption which then fix the amount of resin needed. If the amount of material needed for proper stability is too high, the use of AF should be discarded in favor of a more economical means of stabilization.

The findings of this investigation agree closely with those of Winterkorn (24). Particularly significant is the agreement that the mid-range clay content soils derive the greatest benefits from the resin treatment and that the resin is both a waterproofing agent and a binding agent. The results of the present study also indicate that a minimum of about 2 percent AF is needed for resistance to weathering. Since Winterkorn's studies, which were performed on a variety of soils other than loess, and the loess studies agree so closely, it is plausible to assume that the results may be extrapolated to still different soils with a reasonable degree of accuracy.

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APPENDIX

The order of mixing aniline, furfural, and water into soil has an important influence on the final stabilizing effectiveness of the AF treatment.

The determination of moisture content in samples containing unreacted chemicals is a difficult problem and is considered because of its obvious influence on the moisture-density relationships.

Ratio of Aniline to Furfural

A study of the effects of the ratio of aniline to furfural was made in which this ratio was varied from 1:4 to 3:1. The total amount of aniline and furfural was held constant at 5 percent and mixed with soil 20-2 to have a common basis for comparison. The dry and soaked unconfined compressive strengths were determined along with the total volatile loss during drying and the moisture absorption during soaking.

The data in Table 7 indicate that a weight ratio near 2 parts aniline to 1 part furfural is an optimum ratio. Plots of the data indicate that maxima or minima occur slightly below the 2:1 weight ratio. These results agree closely with those of other investigators. A weight ratio of 1.93:1 is a 2:1 mol ratio which is the exact stoichiometric relation between aniline and furfural as proposed by Zincke and Muhlhausen (27) and supported by Winterkorn (25). Ratios as low as 3:2 and as high as 5:2 could be used without a drastic departure from optimum conditions.

Order of Mixing

Batches of soil in which all possible combinations of the order in which the chemicals and the water could be introduced were mixed. The mixtures were molded and air cured for 10 days and then tested for dry unconfined compressive strength, soaked compressive strength, deformation at failure when tested dry and after soaking, water absorption, swelling, loss of volatiles and shrinkage during air curing. The results were tabulated and an arbitrary value of 10 was assigned to the best performance in each test, 9 to the second best, 8 to the third, and so on. The results of the first five tests were used as evaluation criteria because the results of the last three tests did not vary significantly.

The ratings revealed that the mixing order of water, furfural, and then aniline produced the best product. The second best mixing order was found to be furfural, water, and aniline. Mixing furfural in last produces fair results whereas mixing water in last was shown to be poor procedure.

The results are only slightly better when water is mixed in first than when furfural is mixed in first. It is important that the aniline be mixed in last. This can probably be explained by the fact that furfural is more soluble in water than aniline and furfural is also absorbed by clay to a greater degree than aniline (13).

All mixtures were thereafter made by mixing the water and furfural simultaneously into the soil until the mass was ho-

TABLE 7
 ANILINE-FURFURAL RATIO STUDY DATA

Weight Ratio of Aniline to Furfural	Dry Strength, lb	Wet Strength, lb	Weight Loss during Curing, %	Moisture Absorption after 24-Hr Immersion, %
1:4	1530	190	17.3	15.3
1:2	1630	390	16.6	14.2
2:2	1310	400	15.5	16.1
3:2	2430	1090	15.2	2.9
4:2	2790	1240	15.4	2.2
5:2	2540	1070	15.3	2.6
6:2	2100	580	15.7	10.7

mogeneous. Aniline was mixed in last, just prior to molding.

Determination of Moisture Content

The moisture content of soil is defined as the ratio of the weight of water contained in the soil to the weight of the dry soil: it is expressed as a percentage and is determined by drying a sample to equilibrium moisture content in an oven at 100-110 deg C. The difference between the weights of the sample before drying and after drying is taken as the amount of moisture contained in the soil.

Moisture determination in samples containing unreacted aniline-furfural presents a difficult problem, since aniline and furfural are both volatile. Some loss of these chemicals occurs along with the normal loss of water, although the resin forming reaction begins immediately on contact between the aniline and furfural. The rate of resin formation is dependent upon many factors including temperature and catalysis, whereas the rate of volatilization is dependent mainly on temperature and the amount of unreacted material present. Research has indicated that the loss of unreacted chemicals is also dependent upon the amount of water present at the time of mixing.

The weight of the material after oven-drying includes both the original amount of soil and the residual aniline and furfural that have reacted to form the non-volatile resin. Since varying percentages of resin were studied, all moisture contents for molding purposes were computed as a percentage of the weight of the oven-dry soil. This places the moisture contents at the time of molding on a common basis and permits a comparison of all mixtures, including those composed of only soil and water.

After air curing most of the original mix water has been lost through evaporation and the residue is soil, hygroscopic moisture, and aniline-furfural resin. Moisture is absorbed when treated specimens are immersed or otherwise brought into contact with water or water vapor. Since this moisture is apparently absorbed by both the soil and the stabilizing resin the percentage

absorption is computed on the basis of the combined weights of the oven-dry soil and resin.

The moisture content of specimens molded from a prepared batch may be determined by adding the hygroscopic moisture of the soil used and the amount of water incorporated into the mixture. This method of moisture determination is subject to the errors of evaporation during mixing, evaporation during the process of molding, and the limitations of quantitative measurements prior to mixing. A correction was introduced to compensate for evaporation loss during mixing by adding a predetermined amount of extra water to the mass before mixing. Evaporation during molding was kept at a minimum by covering the mixing bowl containing the batch of soil, water, and aniline-furfural with a water saturated cloth. This keeps the partial pressure of the water vapor in the atmosphere above the mixture at a maximum and evaporation from the mixture at a minimum. Errors due to volumetric apportionment of fluids are easily kept within limits of $\frac{1}{2}$ gm. Since the magnitude of the soil mass is over 1000 gm, the error due to volumetric measurement is less than 0.05 percent of the soil weight.

As a check on the accuracy of this method for moisture determination, some tests were made in which small individual samples of 2:1 AF water-soil systems were oven dried at 100-110 deg C. The soil was first weighed in a small aluminum weighing can after which furfural was added and mixed into the soil. The can and contents were again weighed to determine the amount of furfural added. Water was then added and mixed with the soil and furfural. The can and total contents were again weighed. Finally aniline was added and mixed into the system, the total amount was again weighed and placed in an oven for 24 hours. All of these weighings, together with a separate determination of hygroscopic moisture in the soil, permitted an accurate material balance to be made. The oven dried sample was weighed, and the amount of aniline-furfural lost was determined.

A number of similar samples were pre-

pared, varying the amount of aniline-furfural and the amount of water in each sample. It was found that a plot of the percentage of the aniline-furfural lost in drying vs the weight ratio of initial amount of water to initial amount of aniline-furfural was very close to a straight line with a slope of one and an intercept of 17. The equation of this line permits the derivation of a moisture content equation dependent only on the variables of total loss after oven-drying and the total residue after oven-drying.

Ogilvie (18) made a similar study and found that the moisture may be determined, without appreciable error, by expressing the total volatiles lost as a percentage of the total residue. This was subject to the condition that the moisture sample be held in a covered can for 30 minutes before placing in the oven. The 30-min period appears to

be sufficient for the aniline and furfural to react to a degree that minimizes losses in the oven or at least makes the losses consistent. The previous method requires samples to be placed in the oven immediately.

Both of the above methods of moisture determination are time consuming. Samples containing unreacted AF must be placed in an oven to dry with a consequent volatilization of some of the chemicals. These volatile chemicals have a tendency to be absorbed by other samples and thus produce contamination. The change in weight due to absorbed vapors proved to be serious in some cases. In view of these phenomena, it was decided to use the method of moisture determination in which the moisture is calculated by the amount of water introduced prior to mixing.