Physical Properties of Aggregates Stabilized With Papermill Wastes

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This investigation covers the determination of physical properties of road aggregates stabilized with lignosulfonic acids and lignosulfonic acid polymers, including the effects of heat treatment and the incorporation of hydrated lime. In addition, a limited amount of information on aggregate stabilization by spent sulfite liquor is presented for comparison.

The purpose of this study was to investigate the possibilities of better utilization of sulfite liquor wastes from papermills in stabilizing road aggregates. Because sulfite liquor is water soluble, attempts have been made to produce materials of higher molecular weight from this liquor, thereby decreasing solubility. As a result, lignosulfonic acids and their polymers have been experimentally developed.

The physical properties investigated comprised compressive strength, density, insolubility, and immersion moisture content. Insolubility was determined by the optical density and the Folin-Denis methods.

The lignosulfonic acid polymers investigated were less soluble in water than the lignosulfonic acids. With respect to the compressive strength, relatively good results were obtained with: (a) the heat-treated mixture containing 1 percent lignosulfonic acid polymers without any hydrated lime, and (b) the non-heat-treated mixture containing 1 percent lignosulfonic acids with 3 percent hydrated lime.

For the aggregates investigated, mixtures containing 3 percent hydrated lime without other additives, heat-treated or non-heattreated, showed relatively high values of compressive strength and practically no solubility.

•THE PROPER disposal of spent sulfite liquor (SSL), an industrial waste of papermills, has been a great problem to the paper manufacturers for many years. The great amount of pollution produced by these wastes has caused objections to their disposal into streams and rivers.

As a result of studies made of the proper utilization of SSL, it has been employed extensively as a binder on gravel roads. One of the biggest disadvantages of such utilization is its water solubility. There are three possible ways of alleviating this problem: (a) addition of chemicals such as hydrated lime to form insoluble materials, (b) laying a bituminous mat over SSL-treated bases so that little rainwater will flow through the base courses to dissolve SSL solids, and (c) increasing the molecular weight of SSL by forming lignosulfonic acid polymers, which are relatively insoluble in water.

In recent years, the Sulphite Pulp Manufacturers' Research League at Appleton, Wis., developed lignosulfonic acids (LSA) and lignosulfonic acid polymers (LSAP) from SSL by

an ion exchange method. The most important property of these new materials is their high molecular weight as compared to SSL. It is believed that as the molecular weight of these materials increases, the compressive strength and the insolubility increases. Furthermore, the addition of proper amount of hydrated lime (with or without heat treatment) to LSA or LSAP may produce much improved water insolubility and higher strength, as it has with SSL (1, 2, 4). The application of heat to the stabilized soil samples was suggested by previous research (3).

PURPOSES AND SCORE

The purposes of this study were:

1. To determine the effects on the compressive strength when aggregates were stabilized with various amounts of LSA or LSAP and hydrated lime;

2. To determine the effects on the compressive strength when specimens were heat-treated and then immersed in water;

3. To determine the optical density of solution and solids content produced by the partial dissolution of LSA or LSAP solids from treated specimens immersed in constant volume of water bath for 4 days;

4. To determine the moisture content of specimens at the end of the 4-day immersion in water;

5. To determine the effects on the density and optimum moisture content (or molding moisture content) of stabilized specimens; and

6. To compare these physical properties of LSA and LSAP with the corresponding properties obtained for SSL and hydrated lime.

PROPERTIES OF MATERIALS

Aggregates

The quarry from which all aggregates were obtained is located in the Platteville formation. The lower beds are of the Pecatonica member and the upper thinner beds of the McGregor member. The aggregates are primarily dolomitic limestones. This type of aggregate was selected as typical of aggregates used for base and wearing courses in eastern and southern parts of Wisconsin where LSA and LSAP materials would be available if manufactured in quantity by papermills.

The aggregate properties are given in Table 1, and the design gradation which meets the gradation specification for crushed stone base course of the State Highway Commission of Wisconsin is shown in Figure 1.



Figure 1. Design gradation.

TABLE 1									
PROPERTIES	OF	AGGREGATES							

Property	Value
Coarse aggregates, >2.0 mm (% by wt)	76
Coarse sand, 2.0-0.42 mm (% by wt)	8
Fine sand, 0, 42-0, 074 mm (% by wt)	5
Silt and clay, 0.074-<0.005 mm (% by wt)	11
Eng. classif.	A-2-4
Liquid limit ^a	17.4
Plastic limit ^a	NP
Bulk spec, grav, ^b	2.570
Bulk spec. grav., saturated surface	2,660
Apparent spec, grav,b	2,830
Bulk spec, grav. ^C	2.860
Adsorption, R-4 material (\$)	3.27
pH	8.0
Organic content	Negative

^aAtterberg limits. ^bMaterial retained on No. 4 sieve. ^cMaterial passing No. 10 sieve. TABLE 2 COMPOSITION OF LIME

Percen			
1.2			
0.5			
0.7			
1.6			
54,0			
41.9			
0.5			

Hydrated Lime

The hydrated lime is designated as special finishing hydrated lime of ASTM Designation C 207-49 and meets Federal Specification SS-L-351 Type M

(Masonry). It is dolomitic hydrated lime with a specific gravity of 2.358 and a plasticity of 0.300. Its composition is given in Table 2.

LSA and LSAP

The properties of LSA and LSAP are as follows:

- 1. Strong organic acid;
- 2. High dispersion properties;
- 3. Adhesive properties increase with increasing molecular weight;
- 4. Cationic ion exchange activity;
- 5. Strong reducing agent;
- 6. LSA at high molecular weight form gels;
- 7. LSA react with trivalent metals (Cr^{+++}) ;

8. LSA act as sequestering and chelating agents for many metals, especially iron and copper;

9. Viscosity of clay mixtures decreases with increasing molecular weight of LSA up to a certain limit (relative molecular weight of about 20,000), and then increases with further increase in molecular weight (Fig. 2); and







Figure 3. Relative molecular weight: compressive strength relationship of iron ore briquettes.

10. Compressive strength of iron ore briquettes stabilized with LSA increases with increasing relative molecular weight (Fig. 3).

Additional data are given in Table 3.

SSL

SSL used in this investigation is of softwood origin and produced by calcium base process. Its solids content is 11.0 percent. The composition of the SSL solids is given in Table 4.

An analysis of the dried lignin powder used in this study is given in Table 5. The compound has a specific gravity of 0.31 and a pH in 10% solution of 4.3.

1	PROPERT	FIES	OF	LSA	AND	LSAI	2
.cid	pH	Sol ()	ids 6)	Re	educir ars ()	ig ()a	Mc W

TABLE 3

Acid	pН	(%)	Sugars (1) ^a	Wt	
LSA	0.12	51,5	8.50	10,700	
LSAP	0.69	45.0	5.84	22,800	

^aBy wt of solids.

TABLE 4 COMPOSITION OF SSL SOLIDS

Material	Percent
Lignosulfonates	55
Hexose sugars	14
Pentose sugars	6
Sugar acids and residues	12
Resins and extractives	3
Ash	10

TABLE 5

ANALYSIS OF SSL

Material	Percen		
Lignin	54.5		
Total sugar	19.3		
Iron	0.024		
Magnesium oxide	0.27		
Calcium oxide	6.5		
Sodium oxide	0.033		
Sulfated ash	16.3		
Sulfone SO ₂	3.6		
Sulfur trioxide	0.7		
Free sulfur dioxide	0,17		
Total sulfur	6.1		
Volatile acids	1.2		
Calcium lignosulfonate	65.0		
Fe and Al as oxides	0.1		

MOLDING AND CURING OF SPECIMENS

All specimens were molded in CBR molds. Each specimen was compacted in five equal layers with 25 blows for each layer and 18 in. of free fall by a 10-lb rammer. The specimens were cured for 7 days in 50 percent RH at 75 ± 3 F. Three specimens were made and tested for each category and the average of each three values are used.

After air curing, specimens, which were not to be heat-treated, were immersed in water for 4 days, then tested wet for unconfined compressive strength.

Specimens, which were to be heat-treated, were cured at 210 F for 24 hr and then cured again at 340 F for another 24 hr. They were then cooled to room temperature for 24 hr, immersed in water for 4 days, and tested while moist for unconfined compressive strength.

During immersion of the specimens, some of the uncombined SSL and LSA or LSAP solids were dissolved by water. As the amount of dissolved solids increased, the intensity of color of the solution increased to dark brown. Approximately 1 pint of this solution was taken for optical density, solids content, and pH determinations. Some specimens completely disintegrated during immersion and others were flaked on the surfaces. Some specimens maintained their original shapes quite successfully throughout the immersion period.

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Stabi-	N	Iolding M	loisture (%)	Dry Density (pcf)					
lizer (%)	0% Lime	3≉ Lime	6% Lime	9% Lime	0% Lime	3% Lime	6% Lime	9% Lime		
			(a)	LSA Stabi	lizer					
0	6.0	6.8	7.7	8.6	143.0	140.4	138.0	135.0		
1	5.4 7.9		8.6	8.8	139.5	137.0	134.0	131.0		
2	5.4 8.5		9.5	10.7 138.0		134.0	132.0	128.0		
3	_a	9.4	10.6	11.6 <u>a</u>		130.0	128.0	125.0		
4	_a	10.5	11.5	12.5	_a	126.0	124.0	122.0		
			(b)	LSAP Stat	oilizer					
0	6.0	6.8	7.7	8,6	143.0	140.4	138.0	135.0		
1	6.4	6.4 9.4 10.1		11.0	137.0	134.0	132.0	129.0		
2	7.0	0 10.3 11.0		11.9	135.0 130.		128.0	126.0		
3	7.8	11.0	11.7	12.7	131.5	126,5	125.0	122.0		
4	_a	11.9	12.6	13.3	_a	123.5	122.0	120.0		

TABLE 6 MOLDING MOISTURE CONTENTS AND DRY DENSITIES OF MIXTURES

^aSemiliquid mixture.



WEIGHT OF AGGREGATES

Figure 4. Molding moisture content of aggregate-LSA-lime mixture (Groups 1 and 2).



Figure 5. Dry density of aggregate-LSAlime mixture (Groups 1 and 2).





Figure 7. Dry density of aggregate-LSAPlime mixture (Groups 3 and 4).

Figure 6. Molding moisture content of aggregate-LSAP-lime mixture (Groups 3 and 4).

All immersed specimens were capped with plaster of paris for the compressive strength test. The rate of compression was 0.05 ipm.

The molding moisture content for each composition was determined by trial, beginning at the optimum moisture content and reducing the moisture content until a desirable solidity of soil specimen was obtained. This reduction was necessitated by the slight slumping of specimen at the optimum moisture content. These molding moisture contents were generally from 0.5 to 1 percent less than the optimum moisture content for each composition.

The molding moisture content and the corresponding dry densities for the various combinations of lime and aggregates with LSA and LSAP are given in Table 6. These values are also shown in Figures 4 to 7.

In both LSA and LSAP mixtures, molding moisture contents increased with increasing amount of additives, whereas the dry densities decreased with the increasing amount of additives.

DATA AND DISCUSSION OF RESULTS

Results of the various tests are given in Table 7.

Group 1

Specimens were made with aggregates, LSA, and lime and were heat-treated. The compressive strength is shown in Figure 8. Aggregates stabilized with 3 percent hydrated lime and no other additives indicated the highest compressive strength in this group. However, when aggregates were stabilized with both LSA and lime, relatively high compressive strengths occurred where the ratio of LSA to lime content was about 1 to 3 by weight. At this ratio the compressive strengths were nearly the same.

The immersion moisture content in percent by dry weight of specimen is shown in Figure 9. Aggregates stabilized with LSA alone or with relatively large amounts of LSA as compared to the lime present were quite susceptible to water penetration. Therefore, a certain proportion between LSA and lime contents must be observed to reduce or prevent the water susceptibility, consistent with the desired compressive strength.

This susceptibility to moisture was also reflected in the compressive strength. It is recalled that the compressive strengths of specimens with LSA alone or with a

TABLE 7 PHYSICAL PROPERTIES OF STABILIZED MIXTURES^Q

Stabi-	Comp	ressive	Strengt	h (psi)	Immersion Moisture (%)			Optical Density			Stabilizer Solids (gm/1) ^b					
lizer (≸)	0≸ Lîme	3≴ Lîme	6% Lime	9≸ Lime	0≸ Lime	3≸ Lime	6% Lime	9≸ Lime	0% Lime	3% Lime	6% Lime	9≱ Līme	0≸ Lime	3% Lime	6% Lime	9≴ Lime
						(a)	Group	1: LSA 5	Slabilize	r ^c						
0	D	242	218	193	D	6_9	7.4	8.2	0	0	0	0	0	0	0	0
- 1	41	146	134	100	10.0	7.2	7.7	8.3	30 7	6.3	30	2.6	3.8	0.8	0.4	0.7
2	40	114	148	126	10.0	7.2	7 6	8.2	53_6	20.7	8.3	5.2	6.6	2.6	1.0	1.6
- 3	PD	18	109	142	PD	11_0	8.8	7.9	49_2	53.5	19.3	10.0	6.1	6.6	2.4	1.2
4	—	13	80	96	0.00	11_{-5}	9.0	8.,6	-	45.7	21_1	17.6	-	$5_{*}7$	$2_{*}6$	2.2
						(b)	Group	2: LSA 8	Stabilize	n ^d						
0	D	280	212	114	D	7.8	8.5	9.1	0	0	0	0	0	0	0	0
1	D	163	156	112	D	7.9	8.5	8.7	26.6	12,9	2.7	2.4	3.3	1.6	0.3	0.3
2	D	125	110	81	D	0.4	8.8	9.5	49.8	11,9	6.7	5,1	6.2	1.5	0_8	1.6
3	-	107	96	73	-	9.2	9.2	10.5	-	25.0	10_4	11.4	_	3.1	1.3	1.4
4	-	57	98	80	-	9.7	9.8	9_9	-	34.1	13.6	$12_{+}6$	-	4.4	1.7	1 " 6
						(c)	Group 3	: LSAP	Stabilize	erc						
0	D	242	218	193	D	6.9	7.4	8.2	0	0	0	0	0	0	0	0
1	180	122	125	90	6.8	8.0	8.4	0.0	12.4	2.8	1.4	2.0	1.5	0_4	0.2	0.3
2	122	85	96	84	6.8	8.5	9.0	9.8	22.1	7.6	3.6	2.1	2.7	0_9	0.5	0_3
3	92	52	80	74		10.5	9.6	10.2	19.8	23.7	4.0	3.9	2.5	2_9	0.5	0.5
4	-	28	50	57	- 40	11 ± 5	102	10.4	-	26.2	8.9	5.0	-	3.3	1.1	0.6
						(d)	Group 4	: LSAP	Stabilize	erd						
0	D	280	212	114	D	7.8	8.5	9.1	0	0	0	0	0	0	0	0
1	45	113	109	80	9.7	8.7	9.0	8.9	7-9	2.2	0.9	1.0	1.0	0.3	0-1	0.1
2	22	95	94	64	9.7	9.0	9.0	9.7	24.6	5.4	5.4	2.5	3.1	0.7	0.3	0.3
3	_	62	71	58	-	10.4	10.4	10.5		8.5	5.9	4.1	0.005	1.1	0.7	0.5
4		44	62	51	-	11,5	11_{-4}	11.2	-	16.5	7.5	2 "9	-	2.0	09	0.4
						(e)	Group	5: SSL S	Stabilize	r						
0.9d	D	140	88	77	D	7.6	8.3	9.0	10.0	3.1	2.7	2.7	1.3	0.4	0.3	0.3
2de	D	140	133	120	D	7.4	7.7	8.3	36.4	8.7	4.4	4.0	4.5	1.1	0.6	0.5
200	Ď	118	141	94	D	6.8	7.5	8.3	40.3	9.4	6.5	4 0	5.0	1.2	0.8	0.6

^aD denotes disintegration of specimen in water; PD denotes partial disintegration in water. ^bEstimated by Folin-Denis method. ^cHeat-treated. ^tNon-heat-treated, ^cDried lignin.



Figure 8. Compressive strength of aggregate-ISA-lime mixture (Group 1).



of Figure 9. Immersion moisture content of aggregate-LSA-lime mixture (Group 1).



Figure 10. Optical density value of solution of aggregate-LSA-lime mixture (Group 1).

large amount of LSA were very low due to the high immersion moisture contents and subsequent surface flaking.

Estimates of LSA solids were made by means of the optical density at 282 mµ wavelength. Lignin content cannot be measured accurately because the exact structure of lignin is not known. For this reason, the results obtained by both the optical density and Folin-Denis methods should be considered very rough approximations, based on experience with similar products. The results of optical density and solids content determinations are shown in Figure 10. The amount of LSA solids dissolved in water, as measured by optical density and Folin-Denis methods, increased with increasing amounts of LSA (with one exception), but decreased considerably with increase in lime content for each LSA content. With zero percent LSA, the solution showed zero value of optical density for the lime contents between 3 and 9 percent.

Group 2

Specimens were made with aggregates, LSA, and lime and were not heat-treated.

Aggregates stabilized with 3 percent lime and no other additives indicated the highest compressive strength. When ag-

gregates were stabilized with both LSA and lime, the combination of 1 percent LSA and 3 percent lime indicated higher compressive strength than other combinations of LSA and lime (Fig. 11).

The immersion moisture content is shown in Figure 12. Without the addition of lime, the specimens disintegrated in water, but the addition of only 3 percent lime prevented disintegration. With further increase of lime, the immersion moisture content remained fairly constant for each LSA content.

The optical density increased considerably with the increasing amount of LSA, and decreased with the increasing lime content. For all LSA contents, this reduction in optical density was considerable when 0 to 6 percent lime was added, but beyond 6 percent lime content, the reduction was almost negligible.

Group 3

Specimens were made with aggregates, LSAP, and lime and were heat-treated. Aggregates stabilized with 3 percent hydrated lime and no other additives indicated the highest compressive strength in this group (Fig. 14). On the other hand, when aggregates were stabilized with both lime and LSAP, the combination of 1 percent LSAP and zero percent lime indicated higher compressive strength than other combinations of LSAP and lime. Addition of lime definitely decreased the compressive strengths of all LSAP contents.

The immersion moisture content in percent by dry weight of specimen is shown in Figure 15. Lime was effective in reducing the moisture absorption for 3 and 4 percent LSAP contents. For low LSAP contents (1 and 2 percent), moisture absorption increased slightly with increasing lime content. The highest compressive strength occurred where the immersion moisture content was the lowest.



Figure 11. Compressive strength of aggregate-LSA-lime mixture (Group 2).



Figure 13. Optical density value of aggregate-LSA-lime mixture (Group 2).



Figure 12. Immersion moisture content of aggregate-LSA-lime mixture (Group 2).



Figure 14. Compressive strength of aggregate-LSAP-lime mixture (Group 3).



Figure 15. Immersion moisture content of aggregate-LSAP-lime mixture (Group 3).



Figure 17. Compressive strength of aggregate-LSAP-lime mixture (Group 4).



Figure 16. Optical density value of aggregate-ISAP-lime mixture (Group 3).



Figure 18. Immersion moisture content of aggregate-LSAP-lime mixture (Group 4).



aggregate-LSAP-lime mixture (Group 4).

The optical density or dissolved solids decreased considerably with the increasing lime content-and decreasing LSAP content. In general, beyond 6 percent lime, no significant reduction in optical density was observed (Fig. 16).

Group 4

Specimens were made with aggregates, LSAP, and lime and were not heat-treated. Aggregates stabilized with 3 percent lime and no other additives indicated the highest compressive strength in this group (Fig. ^c17). When aggregates were stabilized with both LSAP and lime, the combination of 1 percent LSAP and 3 percent lime indicated higher compressive strength than any other combinations of LSAP and lime. Further increase in lime or LSAP content was detrimental to obtaining high compressive strength.

The immersion moisture content in percent by dry weight of specimen is shown in Figure 18. Lime was effective in increasing resistance to water penetration. However, lime content greater than 3 percent may not be necessary, because the reduction in immersion moisture content was almost negligible.

The results of optical density and solids content determinations are shown in Figure

19. Lime was effective in reducing the amount of LSAP solids dissolved in water. Lime content greater than 6 percent may not be necessary.

Comparison of Groups 1, 2, 3, and 4

In all cases investigated in this study, aggregates stabilized with lime and no other additives indicated higher compressive strength than others stabilized with both LSA (or LSAP) and lime for corresponding lime content. Aggregates stabilized with 3 percent lime without any other additives indicated the highest compressive strength.

Comparing the compressive strengths of heat-treated specimens of LSA and LSAP, the strength of LSAP without lime was higher than other combinations of additives. If any lime was to be added at all, the compressive strengths of LSA with lime were higher than those of LSAP with lime. With non-heat-treated specimens, the strength of 1 percent LSA at 3 percent lime was higher than other combinations of additives.

The addition of lime to the specimen prevented the disintegration of specimen in the water. Addition of 3 percent lime considerably decreased the immersion moisture content, except in one case. Lime content of greater than 3 percent did not improve the moisture resistance of specimens. The optical density and solids content values of LSA specimens were higher than those of LSAP specimens at all lime contents, regardless of heat treatment. In other words, LSA solids were more soluble in water than the LSAP solids at all lime contents, regardless of heat treatment.

These observations confirm the earlier statement that as the relative molecular weight of LSA increases up to about 20,000, the insolubility of LSA solids increases. As indicated earlier, the relative molecular weight of LSA is 10,700 and that of LSAP is 22,800.

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Figure 20. Compressive strength of aggregate-SSL-lime mixture (Group 5).

Comparison of Aggregate Stabilization by SSL, LSA, and LSAP

A limited amount of pertinent data on the compressive strength, immersion moisture content, and optical density of aggregate-SSL-lime mixture are shown in Figures 20, 21, and 22, respectively, for comparison with the results of LSA and LSAP aggregate stabilization.

Figures 8 and 20 (heat-treated) indicate that the maximum compressive strength attained for 2 percent solids of SSL and LSA was nearly the same at 6 percent lime content. It is noted, however, that at zero percent lime content, LSA specimens had some strength and maintained their shape fairly well in water, whereas SSL specimens disintegrated completely in water.

In Figures 14 and 20 (heat-treated), the maximum compressive strength for 2 percent solids of LSAP was lower than that of SSL for the lime contents of 3, 6, and



Figure 21. Immersion moisture content of aggregate-SSL-lime mixture (Group 5).



9 percent. However, at zero percent lime, LSAP showed considerable compressive strength in contrast to the disintegration of SSL specimens in water.

Comparison of Figures 11 and 20 (non-heat-treated) indicates that the compressive strength of 1 percent LSA was higher than that of 0.9 percent SSL solids at all lime

contents. However, for 2 percent solids, the compressive strength of SSL was higher than that of LSA at all lime contents. Without lime, all specimens of both SSL and LSA disintegrated in water.

Figures 17 and 20 (non-heat-treated) indicate that the maximum compressive strength of SSL for 2 percent solids was higher than that of corresponding LSAP solids at all lime contents. For 0.9 percent of SSL solids and 1 percent of LSAP solids the maximum compressive strengths of SSL samples were slightly higher than that of LSAP at 3 percent lime, but at 6 and 9 percent lime, lower strength was observed for SSL samples.

Comparison of the optical density values of all five groups indicates that for the corresponding solids content of SSL, LSA, and LSAP, LSAP samples showed lower optical density values than those of SSL and LSA. SSL specimens, however, indicated lower values of optical density than those of LSA (Figs. 10, 13, 16, 19 and 22).

CONCLUSIONS

Based on the materials and methods employed in this investigation, the following conclusions may be stated:

1. Mixtures containing hydrated lime and no other additives, both heat-treated and non-heat-treated, showed relatively high values of compressive strength and practically no solubility. The optimum lime content for high compressive strength was 3 percent.

2. The non-heat-treated mixture containing 1 percent LSA with 3 percent hydrated lime gave higher compressive strength than any other combination of LSA and hydrated lime, both heat-treated and non-heat-treated; this mixture also gave higher compressive strength than any combination of SSL and hydrated lime investigated.

3. The heat-treated mixture containing 1 percent LSAP and no other additives indicated higher compressive strength values than for higher percentages of LSAP, heattreated and non-heat-treated, and for any combination of LSAP and hydrated lime, both heat-treated and non-heat-treated; this 1 percent LSAP with no additive also had higher compressive strength than any combination of SSL and hydrated lime investigated.

4. The insolubility values of LSAP stabilized specimens were generally higher than those of LSA and SSL specimens for corresponding hydrated lime contents, whether heat-treated or non-heat-treated.

5. Hydrated lime was effective in reducing the penetration of water into the specimens of aggregates stabilized with LSA or LSAP. The optimum lime content from the standpoint of resistance to water penetration was 3 percent.

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