

THE USE OF SULPHUR AS A RIGID BINDER AND FOR  
THE IMPREGNATION OF CONCRETE

— STATE OF THE ART —

by

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(The opinions, findings, and conclusions expressed in this report are those of the author and not necessarily those of the sponsoring agencies.)

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## SUMMARY

Recent research has led to the development of durable modified-sulphur mortars, concretes, and coatings. All of the methods of using sulphur as a binder for rigid concrete rely on the reaction of one or more modifiers to stabilize, in the hardened state, at least a portion of the sulphur in its less brittle, less dense form. The durability of the concrete produced appears to depend on the modifying system used. In all cases the sulphur must be heated to a liquid state to react with the modifier and to mix with and coat the aggregate and filler.

Sulphur concrete can develop high strength, attains strength in a few hours, requires no special curing, resists acids and organic liquids, has no known undesirable reactions with aggregates, and requires no limitation on the ambient temperature at the time of placement.

When its use becomes economically feasible, sulphur concrete will be an excellent material for use in pavement repairs and bridge deck overlays.



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BACKGROUND

Sulphur is the fourteenth most common element in the earth's crust. Elemental sulphur occurs in small amounts as precipitations from the gases of fumeroles and volcanos and in thermal springs. Large deposits of pure sulphur have been formed by nature in salt domes by the reduction of gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ).<sup>(1)</sup> Substantial amounts of sulphur are also found in the sulphides of iron (pyrite), lead (galena), zinc (sphalerite), mercury (cinnabar), and in the sulphates of calcium, magnesium, strontium, and barium.

Sulphur was one of the first elements known. The ancient alchemists puzzled over its properties and used it in medicine and magical concoctions. Today it is a very necessary raw material for the chemical, medicinal, and agricultural industries. Large quantities are required for the vulcanization of rubber, in the wood pulp industry, and for use as fertilizer.<sup>(2)</sup> It is only recently that sulphur has been in sufficient supply to consider its use as a construction material. Until the early 1970's it was assumed that most of the world's sulphur would always be obtained from deep salt dome deposits by the Frasch process of melting and forcing sulphur to the earth's surface through concentric pipes with superheated water.<sup>(3)</sup> Certain amounts of sulphur have always entered the market as by-products of the mining and refining of the sulphide ores of zinc, copper, and other nonferrous metals.

Sulphur is now being produced as a by-product of the fuel gas industry. The energy shortage has prompted the use of fuel gas from sources high in hydrogen sulphide ( $\text{H}_2\text{S}$ ) and sulphur dioxide ( $\text{SO}_2$ ) (sour gas). Sulphur gases must be removed from the fuel before transport. In 1968 a number of fuel gas processing plants opened in Alberta, Canada, and by late 1982 it is expected that numerous such plants will be operated in the United States.<sup>(4,5)</sup> By 1974

the Alberta sour-gas plants had accumulated 11 million tons of sulphur as a by-product. It was expected that this inventory of sulphur would double before 1977. Sulphur produced as a by-product is called "involuntary" sulphur, whereas sulphur obtained by the Frasch process is termed "voluntary."

In 1972, "The First Annual Report" of the Secretary of the Interior delineated the problems related to the oversupply of sulphur, and predicted that by 1980 the supply in the United States would exceed projected demand by about 23 million short tons.<sup>(16)</sup> Prompted by this annual report and the results of its own research on the processing of sour gas, the United States Bureau of Mines began a research program on the uses of sulphur. The Sulphur Development Institute of Canada and Sulphur Innovations, Ltd. undertook similar research.

It has been found that modified elemental sulphur can be used as a hot liquid cement to completely replace the water and portland cement in ordinary concrete.<sup>(4,7,8,9,10,11,12)</sup> This product is called sulphur concrete. Also, liquid sulphur can be used to impregnate portland cement concretes fabricated with a high water-cement ratio. This allows a reduction in the amount of portland cement used.<sup>(13)</sup> Beyond the scope of this report but of interest to highway technologists is the fact that sulphur has been successfully used in flexible pavements as an asphalt extender and asphalt substitute. "Sulphur-81 an International Conference on Sulphur" held in Canada in May 1981 included four sessions on the uses of sulphur in asphalt and as an asphalt substitute.<sup>(14)</sup>

#### PROPERTIES OF SULPHUR

The ancient word for sulphur, brimstone, means rock that burns. Not only does sulphur melt and burn at fairly low temperatures, but it exists in nine forms or allotropes. The commonest mineral form is orthorhombic ( $S_{\alpha}$ ). Two monoclinic forms ( $S_{\beta}$  and  $S_{\gamma}$ ) and one polymeric form ( $S_p$ ) occasionally occur in nature; however, they are metastable and in time recrystallize in the orthorhombic unit cell. The molecule of sulphur is, in general, a ring of eight atoms, with each sulphur atom being attached to two others. In the polymeric form the rings have become long chains of molecules without any regular arrangement between chains.<sup>(15,16)</sup> This is commonly termed "amorphous sulphur." Sulphur melts at 234°F (113°C) to 248°F (120°C), depending on the form, and becomes a yellow liquid. Between 356°F (180°C) and 500°F (260°C) liquid sulphur partially polymerizes and becomes dark red and viscous. In the presence of oxygen, sulphur will ignite at about 500°F (260°C) and produce poisonous  $SO_2$  gas.

Early attempts to cast useful items from sulphur or use sulphur as a cement with various aggregates were hindered by the fact that the sulphur took its original shape in one of the less dense allotropes or a mixture of them. In time, sometimes many months, and hastened by fluctuations in the temperature, the sulphur reverted to the denser orthorhombic unit cell with concomitant shrinkage and cracking. In the orthorhombic form sulphur is brittle and has one-half the tensile strength of newly cooled sulphur.<sup>(16)</sup> Orthorhombic sulphur is also subject to deterioration by bacteria, sunlight, very strong alkalies, and thermal fluctuations.

### SULPHUR CONCRETE

The intensified research of the 1970's led to the development of commercially feasible, durable, modified-sulphur mortars, concretes, and coatings by the Sulphur Development Institute of Canada in conjunction with Chevron Chemical and Chevron Research, by Sulphur Innovations Ltd. of Calgary, by the University of Calgary Interdisciplinary Research Group, and by the United States Bureau of Mines in conjunction with Chemical Enterprises, Inc., of Houston and Odessa, Texas.

All of the methods of using sulphur as a binder for rigid concrete rely on the reaction of one or more modifiers to stabilize at least a portion of the polymeric form of sulphur in the hardened state. These reactions prevent the recrystallization of the sulphur into its less dense form; they prevent the loss of internal integrity and, thus, the loss of strength and durability.<sup>(17,18)</sup>

In all cases the sulphur must be heated to a liquid state to react with the modifier and to mix with and coat the aggregate and filler. Most systems require a dense-graded aggregate and, usually, a mineral filler such as fly ash or limestone dust. Such a grading minimizes the amount of sulphur required.<sup>(19)</sup>

Sulphur concrete can be mixed in asphalt batch plants. The aggregate must be dry and heated to 375°F (190°C) if mixed with solid sulphur, or 325°F (163°C) if it is to be mixed with liquid sulphur,<sup>(19,20)</sup> Liquid sulphur must be kept at about 285°F (140°C). The completed mix can be transported to the point of placement in a specially heated (275°F {135°C} to 325°F {163°C}) rotating concrete transit mixer. By using sufficiently heated aggregate, the entire mixing procedure can be done in the heated transit mixer. Consolidation is by internal probe vibrators and by vibrating screeds. Hand tools and screed may be wood or metal, but metal tools must be kept hot to prevent adherence of the sulphur.

Proprietary modifiers have been developed by the Sulphur Development Institute of Canada and by Sulphur Innovations. They are designed to be added to the sulphur during the mixing with the heated aggregate in a one-step process.

The United States Bureau of Mines has developed the use of "mixed modifiers" in which the chemicals dicyclopentadiene (DCPD) and oligomers of cyclopentadiene (O-CPD) can be used in varying amounts depending on the properties desired. Sulphur containing 5% of a half and half mixture of these modifiers produces a rigid concrete superior to portland cement concrete in many respects.<sup>(18)</sup> The combination of chemicals is necessary to control the reaction between the sulphur and the modifiers and to assure that the concrete has low permeability and good freeze-thaw resistance. This modifier-mixture must be reacted with the sulphur in a pressurized vessel for at least four hours. After the reaction is complete, the liquid-modified sulphur may be mixed with dense-graded aggregates and fillers and placed, or it may be cooled for storage or shipping in a granulated or prilled form. If cooled, the modified sulphur need only to be mixed with hot aggregate for less than a minute to form concrete. Properly made, such concrete is strong, durable, and nearly impermeable. It has good resistance to fatigue, excellent resistance to acid, and attains 80% of its full strength in the time it takes to cool.<sup>(4,7,8,9,10,11,12)</sup> Table 1 compares some of the properties to the two main types of sulphur concretes with the properties of portland cement concrete.

#### SULPHUR IMPREGNATED CONCRETE

In 1975 Malhotra reported on research with sulphur-infiltrated concrete and noted that sulphur has economical advantages over organic polymer.<sup>(27)</sup> When the impregnation was vacuum assisted, an exceedingly strong and durable concrete could be produced by precasting. Malhotra did not recommend his particular formulation for cast-in-place use. In effect, the sulphur was used as a substitute for some of the portland cement, because the concrete, being infiltrated, had a water-cement (w/c) ratio of 0.80, about 4.3 bag/yd.<sup>3</sup> In 1975 it was concluded that at least in Canada this infiltrated concrete would probably be less costly than portland cement concrete having a w/c of 0.47. The sulphur-infiltrated concrete had a compressive strength of over 10,000 lb./in.<sup>2</sup> (700 kg/cm<sup>2</sup>), withstood 800 cycles of freezing and thawing without damage, and was exceedingly resistant to chemical attack. Apparently, the sulphur fills the capillaries and prevents absorption of water or chemicals and this prevents the critical saturation that causes freeze-thaw distress. A personal communication from Malhotra indicated that there had been no change in the properties of this laboratory concrete in five years.<sup>(28)</sup>

Table 1. Comparison of Portland Cement Concrete with Sulphur Concretes

Property	Portland Cement Concrete w/c 0.47		Sulphur Concrete with Proprietary Modifiers		Sulphur Concrete with Binder Modified with DCPD 2 1/2% and 0-CPD 2 1/2%		
	Average				Aggregate: Basalt	Limestone	Quartz
Strength					5090 psi	9020 psi	6120 psi
Compressive	4500 lb./in <sup>2</sup>	31 MPa	4000 to 10152 psi	27.5 to 70 MPa	35 MPa	62 MPa	42 MPa
Flexural	675 psi	4.7 MPa	1740 to 2900 psi	12 to 20 MPa	950 psi 6.5 MPa	1630 psi 112 MPa	1330 psi 9 MPa
Tensile	450 psi	3.1 MPa	435 to 1160 psi	3 to 8 MPa (21)	860 psi 6 MPa	895 psi 6 MPa (22)	790 psi 5.4 MPa
Absorption	2-4-5%		1.5% (21)		0.1% (17)		
Time to 80% final strength	28 days		A couple of hours more or less depending on ambient temperatures and mass of placement, i.e. time to cool to solidification.				
Limitation on ambient temperature at placement	Yes		None (21)		None (17)		
Curing procedure	Required		None		None		
Moisture in substrate	Moisture OK Puddles prohibited		Hot sulphur cement must not come in contact with moisture. A vapor barrier such as plastic sheeting can be used if base cannot be dried. (23)				
Reactions with reinforcement	Deleterious if water and salt reach the steel		Coated rebars required as even this low absorption allows formation of acid at the steel		No deterioration seen under severe test. (24)		
Reaction with old PCC	Good bond can be achieved. No reactions		Good bond is easily achieved.		Research is needed on possible reactions over long time weathering conditions. (21)		
Chemical reactions	Alkali silica Alkali carbonate		None (6)		None (22)		
Prohibited types of aggregate	Several including excess fines, poor particle shape, almost all clays		Swelling clays are the only prohibited aggregates. Grading should be dense, with 6% to 10% passing 200 mesh. May use many aggregates unsuitable for PCC. (10,18,19)				
Deterioration in sulfate or acid ground water or seawater	Frequent		No deterioration has been detected under severe test. (8,22,23,24)				
Resistance to industrial chemicals	Poor acid resistance		Very good acid resistance (18,21)				
Freeze-thaw resistance	Good with proper air entrainment		Poor — improvement is probable with certain not yet available proprietary additives (8,26)		Good — relative dynamic modulus above 75% at 300 cycles. Probably due to low absorption. (17,18)		
High temperature reactions	At very high temperatures may develop cracks. Will not burn		Will soften if the concrete reaches 223°F (106°C) and will then lose strength. Will not support combustion although surfaces exposed to flame will char and scar. Safety codes may limit use in structural members and in dwellings.				
Bacterial attack	None		Attacked by thiobacillis and thiooxidans forming sulphurous acid and sulphuric acid causing concern for environment (21)		None detected under test (24)		

Work by Brown and Baluch with concretes of a lower w/c ratio soaked in a molten sulphur bath has indicated that some sulphur-infiltrated concrete lacks durability when exposed to certain temperature changes and that it can be seriously attacked by water, acids, and salt solutions.(29)

In 1979 Malhotra stated that sulphur-infiltrated concrete (w/c of 0.70) can have compressive strengths of 14,500 lb./in.<sup>2</sup>, flexural strengths of 1,450 lb./in.<sup>2</sup>, high resistance to acid, and, usually, a satisfactory freeze-thaw durability.(21) It may be unstable in alkaline solutions and when submerged in water for a long period of time. These concretes are not to be considered a substitute for general use, conventional concrete, but in extremely harsh environments sulphur-impregnated concrete will probably have two or three times the life expectancy of ordinary concrete.

In the late 1970's, sulphur modifiers were developed which retain most of the good properties of newly cooled sulphur for a very long time. Yuan impregnated two concretes, clay bricks, and masonry blocks with modified sulphur.(13) He reports that compared to conventional concrete the modified sulphur provided better mechanical, chemical, and physical properties; had a higher reduction in water absorption; exhibited no disintegration or cracking with water storage; and showed better acid resistance and better protection for reinforcement bars.

#### COMPARATIVE ENERGY CONSUMPTION

##### Portland Cement Concrete

According to Halstead,(30) a lane mile 1 in. thick of average ordinary portland cement concrete consumes the following energy expressed in equivalent gallons of diesel fuel (139,000 Btu/gal.):

	<u>Gal./mi-in.</u>
Processing of aggregate	189
Processing of cement	5,723
Transportation of cement	200
Transportation of aggregate - short haul (10 miles)	224
long haul (100 miles)	2,246
Batching concrete	160
Transportation of concrete - short haul	336
long haul	1,790
Placing and consolidating concrete	<u>86</u>
Short Haul Total	6,918
Long Haul Total	10,394

Sulphur Concrete (Estimated)

Data similar to those above are not available for sulphur concretes, but the following extrapolations have been made based on Halstead's data for asphaltic concrete and assuming 20% modified sulphur in the batch, an involuntary source of sulphur, (the processing energy was intended to clean a fuel and cannot be attributed to the sulphur), and an aggregate grading like that of asphaltic concrete but 84.2% of the quantity.

	<u>Gal./mi-in.</u>
Processing of aggregate	190
Processing involuntary sulphur	0
Modification of sulphur	
Heat to 250°F + exothermic reaction 153 tons x 1.8 (estimated)	275
No data on energy consumption of modifiers	?
Transportation of modified sulphur (estimated high to include granulation and bagging)	400
Transportation of aggregate - short haul (10 miles)	234
long haul (100 miles)	2,341
Heating aggregate to 350°F	
$\frac{350^{\circ}-70^{\circ}}{300^{\circ}-70^{\circ}} \times \frac{612 \text{ tons}}{727 \text{ tons}} \times 1,404 \text{ gal.}$	1,439
Operating batching plant	109
Transportation of hot sulphur concrete with heaters (Estimated)	
short haul	400
long haul	2,137
Place and consolidate sulphur concrete	86
Short Haul Total	3,133
Long Haul Total	6,977

Comparison

It can be seen that these estimates indicate that for each lane mile inch the equivalent of 3,700 gallons of diesel fuel can be saved by the use of sulphur concrete as compared with portland cement concrete. For short hauls, the sulphur concrete uses 45% as much energy and for long hauls 67%.

Halstead's<sup>(30)</sup> figures for asphaltic concrete total 2,669 gal./mi-in. for short hauls and 6,595 gal./mi-in. for long hauls, exclusive of the energy intrinsic in the asphalt, the calorific energy, 10,220 gal./mi-in. Whether calorific energy should be used in estimates of energy consumption is a matter of discussion and depends upon the quantity of contaminants in the original fuel oil.

Even without including calorific energy, it can be seen that the estimated energy consumption for sulphur concrete is only a little more than that for asphaltic concrete.

#### WASTE AND SURPLUS MATERIALS

Sulphur concrete requires the use of a different grading of aggregate than does portland cement concrete. Ten percent of the aggregate should pass the 200 mesh. Fly ash, silty clays, and the fines from other aggregate preparations can be used for the extra fine portion of the aggregate. There are no known aggregate reactions; thus sulphur concrete can utilize those aggregates that are chemically reactive in portland cement concrete. Waste glass and pottery can be used as aggregates, as can mine tailings and spent oil shale. (17,18)

Research is needed to define the particle shape requirements of aggregate for sulphur concrete, and to discover if any deleterious reactions are possible when recycled aggregate materials contain plaster of paris and portland cement paste.

#### POLLUTION

There is no evidence of any pollution of the environment caused by the presence of sulphur concrete fabricated with properly reacted, mixed-modifier sulphur. The material is nearly impermeable and insoluble and completely untouched by the chemicals and bacteria found in the ground and in the air. (17,22,23,24)

Batching of sulphur concrete in a well-ventilated area and with reasonable temperature control poses no danger from evolution of the noxious hydrogen sulphide and sulphurous oxide gases. (31) No special breathing equipment is required.

The reaction that produces the mixed-modifier sulphur cement is kept at a safe temperature for the sulphur. The reaction occurs in a pressurized vessel, so there is no loss of the modifiers into the atmosphere.

#### SAFETY

As mentioned above, there are no dangerous concentrations of gases present during the production and placing of sulphur concrete in a well-ventilated area.

Sulphur concrete is placed hot but is no more dangerous than asphaltic concrete. The same sort of protective clothing, gloves, and boots would be used for sulphur as for asphaltic concrete.

Modified-sulphur concrete cannot support combustion, but the application of flame will char the surface. Sulphur is a good insulator and it takes a long exposure to raise the temperature of sulphur concrete to 223°F (106°C). If a structural member is so heated, it will soften. Its use, therefore, is not recommended for making load-bearing members. Extremely high temperature can cause the evolution of noxious gases. Because of this danger, sulphur concrete is not recommended for dwellings furnished and decorated with flammable materials.

#### COSTS

Sulphur concrete is at present about twice as costly as portland cement concrete in Virginia, or even closer to a source of sulphur.<sup>(32)</sup> The mixed-modified sulphur cement is available from only one source, which is in Texas. Costs will probably go down as the supply of involuntary sulphur increases. As there is no excess involuntary sulphur in Virginia,<sup>(33)</sup> shipping will add to the cost.

#### USES

Despite its high cost, sulphur concrete has properties that will probably make it economically advantageous in the following situations:

1. Rapid strength gain for patching highly trafficked highways.
2. Low permeability makes it suitable for use in the repair of bridge decks.
3. Lack of reactions with aggregate makes it advantageous for use in regions short of good aggregate for portland cement.
4. Lack of reaction with acid ground water makes it suitable for use in areas high in sulfate, sulphides, and sulphuric acid.
5. Ability to be placed at any temperature provides advantages in extreme weather conditions.

At present, sulphur concrete is being used to fabricate and repair floors in chemical plants where acids and hydrocarbons have quickly destroyed portland cement concrete floors. It is being used to construct vats for the containment of hydrocarbons and strong acids, and for precast items such as sewer pipe, drop-inlets, curb and gutter, and median barriers. A large precasting plant in Alberta has an 8-ton reactor for producing mixed-modifier sulphur concrete of the type developed by the U. S. Bureau of Mines,<sup>(20)</sup> and it is capable of producing several kinds of sulphur cement.

Sulphur is being used in flexible paving, in sulphur-extended asphalt, sulphur-sand asphalt, sulphur-recycled asphalt, and various formulations such as sulphlex.

There are no available data concerning sulphur concrete modified to produce a rigid concrete for use in patching and repairing portland cement concrete. The rapid strength gain, the lack of a need for curing procedures, the low absorption, the good freeze-thaw resistance, and the ability to be placed at any temperature would seem to indicate that mixed-modified sulphur concrete might be an excellent material for such repair. It appears that repairs being made with sulphur-bearing materials are utilizing one of the sulphur-asphalt mixtures.

No data are available on the use of sulphur concrete for bridge deck overlays, although the properties of the material seem to indicate that it should prove durable and watertight. Federal funding for such an experimental bridge deck is likely in the near future.<sup>(32)</sup>

#### RESEARCH NEEDS

Further research is required on certain facets of the use of sulphur concrete. No data are available concerning the reaction, if any, of sulphur concrete with a moist alkaline system such as portland cement under weather conditions. The reactions of sulphur concrete with aggregates fabricated from recycled materials are unknown. No data are available concerning the particle shape required of aggregates used in sulphur concrete, nor is there information on means of securing skid resistance or on the adherence of marking paint.

## RECOMMENDATIONS

It is recommended that any further data collection by the Research Council include on-site inspection of placements and interviews with highway department people involved.

It is recommended that any steps towards the widespread utilization of sulphur in Virginia await the results of research on the use of it as a patching material, a bridge deck overlay, or in other applications. The Research Council should remain alert to any developments in this field.

Unless detailed instructions for the mixing and placing of sulphur concrete become available, it will probably be necessary for the first sizable Virginia placement of this material to be made by a contractor experienced in its use. Such a placement should be made with Virginia aggregates and probably in a zone having a severe climate.



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