Gel Sealants for the Mitigation of Spontaneous Heatings in Coal Mines

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By Yael Miron

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

<u>Metric Units</u>

cm	centimeter	m	meter
g	gram	mL	milliliter
kg	kilogram	°C	degree Celsius
L	liter		

<u>U.S. Customary Units</u>

gal	gallon	pct	percent
lb	pound	s	second

GEL SEALANTS FOR THE MITIGATION OF SPONTANEOUS HEATINGS IN COAL MINES

By Yael Miron[1]

ABSTRACT

Coal mines that undergo self-heatings, and specifically mines that sustain both floor heave and self-heatings, would benefit from the use of sealants to minimize air ingress to susceptible areas. The U.S. Bureau of Mines (USBM) evaluated three sealant systems for this purpose, bentonite-water mixtures, silicate gel, and guar gum gel. The guar gum-based gel was the only sealant that possessed the desired properties of elasticity, ease of preparation, adaptability to the mine, and a long life. In addition, since the gel consists mostly of water, it could quench or cool nearby heatings. Optimal formulations were determined in laboratory tests, and then tested in the USBM Safety Research Coal Mine. Two formulations resulted in stable gels that lasted for at least 12 months in the mine. Semicontinuous preparation of gel using an eductor was evaluated and appears feasible. Although guar gum gel is expensive, selective use to prevent heatings in specific areas would be cost-effective, both in terms of productivity and enhanced mine safety.

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INTRODUCTION

Fires in underground coal mines can result in loss of life and property. Between 1978 and 1988, approximately 15 pct of underground coal mine fires in the United States were attributed to spontaneous combustion (*1*).[2] These fires usually occur in worked out or gob areas of the mine, and are difficult to detect. In general, low-rank coals are more susceptible to self-heating and the resultant spontaneous combustion, than high-rank coals. However, heatings can occur in mines of high-rank coal due to the effects of other parameters such as mining and geological conditions and ventilation rates, among others.

One such mine, mining a high-rank coal, is the Jim Walter Resources (JWR), Inc., No. 5 Mine located in the Black Warrior Coal Basin in north-central Alabama. This mine is the farthest southward into the basin of all four company mines that extract the Blue Creek Coalbed of the Mary Lee Coal Group. The mine began to experience floor heave in 1981. The area of the mine that experienced the floor heave is in a zone characterized as the "expanded Blue Creek Seam" where the coalbed consists of upper and lower benches. In this area, the lower bench has a highly variable strata consisting of interspersed thin layers of coal, shale, and fire clay (*2*). The mine, which produces high-grade bituminous coal, is over 600 m deep. At this depth, the rock is under considerable stress from the weight of the overlying rock.

Severe floor heave caused production delays and required extensive and expensive ground control measures. New pillar design improved ground control, but did not eliminate the heave problem completely (*3-4*). Then in November 1986, a heating was discovered in the floor of the mine. Since then many other heatings have occurred (*5*). All of the heatings have been located in a section of the mine in the vicinity of a fault line, where an "expanded Blue Creek Seam" exists beneath the floor. Analysis of samples from the area beneath the floor showed the presence of varying amounts of finely disseminated pyrite (*6*). Such pyrite is highly reactive and is easily oxidized when the ground heave exposes the material beneath the floor to air and moisture. This oxidation is highly exothermic in nature and, as a result, the pyrite self-heats. The higher temperatures, in turn, accelerate the oxidation of additional pyrite and of the carbonaceous matter in contact with the pyrite (*6*). The end result is a heating, which may progress to a fire if not detected in time. Two heatings have occurred during 1994-95 in the floor of the adjacent No. 4 Mine. These heatings did not occur in the expanded seam area, but did involve pyrite.

The Warrior Basin coalbeds contain large amounts of high-quality methane. Methane drainage ahead of mining is practiced by the mines in the area, including the No. 4 and No. 5 Mines (*7*). This practice helps to reduce methane emissions at the face by as much as 60 to 70 pct. However, high ventilation rates are prevalent throughout many of the mine areas because of the methane hazard and the depth of the mines. These high ventilation rates produce high differential pressures across stoppings and seals, resulting in air leakage and thereby providing ample air for the oxidation of both pyrite and adjacent coal. In large part, the leakage occurs at the junction of the stopping with the strata and through cracks in the strata. To eliminate leakage, the use of balancing chambers is recommended for the short term; however, for the long term, injection of cement or backfill material is preferable (*8*).

To eliminate or reduce the rate of pyrite and coal oxidation in the JWR No. 5 Mine, or in other mines facing similar problems, access of air to areas enriched with pyrite or to areas containing coal that is susceptible to spontaneous combustion, should be minimized. Control of mine floor heave depends on the cause and type of floor heave. Wuest (*9*) describes physical characteristics and mining conditions that contribute to floor heave, as well as procedures for control of heave. The latter include methods of mine maintenance, supplemental support, and mine design and layout. Supplemental support methods include the installation of floor bolts or concrete linings or the injection of polyurethane. These are extreme measures that, depending on the size of the affected area, can become impractical. In addition, high costs of material, labor, and equipment are associated with such procedures (*9*).

Although few mines suffer from such an assortment of problems as the No. 5 Mine, numerous mines encounter at least a few of these problems. Additionally, some floor upheaval is common to many mines. However, when floor heave is extensive and frequent, floor bolts and stiff concrete and grout will not hold up, but instead will crack, break, and become useless. Even backfill material will be shifted away from the area if the magnitude of upheaval is large. In such cases, the only practical choice may be to block access of air to specific, critical areas, and use sealants that are elastic in nature.

A review of the literature regarding the use of sealants in mines for the purpose of reducing occurrences of spontaneous combustion indicates the use of clay mixtures, various cements, and grouting material. However, in most of these cases, floor upheaval is not cited as a problem; rather, it is air leakage around seals and through cracks in the strata that is of major concern.

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[3]Italic numbers in parenetheses refer to items in the list of references at the end of this report.

Bentonite clay has been used by various mines as a grouting material. Bentonite is a montmorillonite-type clay formed by the alteration of volcanic ash. It is highly colloidal and can absorb large amounts of water. Absorption of water is accompanied by a large increase in volume. Bentonite grouting was used at the Agecroft Colliery, in the Doe Seam, in Lancashire, United Kingdom (*10*). This deep mine (640 to 670 m) had to change its working horizon due to a particular geological condition including a steep gradient and the appearance of very hard sandstone immediately above the coal seam. As a result, much coal was left in the roof, and several heatings took place. A permanent grout pumping operation was established in the mine to afford continuous pumping into the gob and, in combination with continuous vigilance, the problem was contained. This mine did not sustain floor upheaval occurrences.

The use of a sealant consisting of a mixture of Portland cement, limestone, exfoliated vermiculite, and other in-gredients, was reported some years ago for a mine in eastern Ohio (*11*). This mine, one of the mines located near the edge of the Pittsburgh No. 8 coal basin, had pyrite and a clayey shale in the roof. Both weathered badly, especially in the moist air of summer, and caused roof falls. Fire clay (a clay predominantly composed of kaolinite, high alumina, or silica that forms the seat earth of many coalbeds), present in the floor strata, also weathered at a high rate and, via its action on the pillars, contributed to further roof falls. The sealant, developed in the United Kingdom, was used successfully to seal stoppings and overcasts, as well as for fireproofing and construction. Mixtures of clay with other ingredients were tried in coal mines in various other countries. In the former Soviet Union (*12*), slurries of clay with sodium silicate were used. In Japan (*13*), clay suspensions and clay-cement mixtures were tried, but the results were not good due to formation of cracks in the sealants. Finally, suspensions of clay with aqueous solutions of calcium chloride ($CaCl_2$) were used in eastern European mines that were experiencing heatings and fires due to spontaneous combustion, with very good results reported (*14*).

Asphalt-based sealants were tested in some mines. Excellent results were cited for an asphalt solution that was sprayed on various surfaces inside a mine (*13*). An asphalt emulsion consisting of 40 pct water and 60 pct semisolid oil asphalt was applied onto samples of shale from the roof of a coal mine that experienced roof deterioration (*15*). Pyrite and other minerals in the roof oxidized, and the resultant roof fall caused spontaneous combustion. The tests in the laboratory with the emulsion and roof material gave good results, and the mine planned to apply the emulsion to the roof with a spray gun and

pressurized air. However, it is not known if this method was actually used by the mine. Good results were also reported for the use of a mixture of tar and burnt oil to prevent spontaneous combustion of coal in quarry benches in India (*16*). However, asphalt- and tar-based coatings or sealants will ignite from contact with fires, so their safe use is questionable.

Sealants based on latex preparations consisting of 60 to 70 pct of finely dispersed rubber in water solutions were evaluated in some mines. Neoprene latex emulsions were sprayed onto mine surfaces in eastern Europe (*14, 17*). Initially, results seemed good, but on drying the sealants shrank and did not perform well.

Finally, sealants based on gel solutions of water glass or other materials are also reported in the literature, usually with good results. Gel solutions of water glass with ammonium sulfate and water were injected into gob areas in Russian coal mines (*18*). A water glass solution was used in the laboratory to coat coal samples from a mine and was found capable of retarding oxygen adsorption by the treated samples (*19*). A pillar in the mine was then infused with the solution and the rate of oxygen adsorption by the pillar was likewise lowered (*20*). A gel-like solution, able to adhere to the roof, rib, and floor in a mine, was prepared from a commercial mixture of "Light Water TM" with other additives (*12*). The elastic gel was applied to a thickness of approximately 1 cm to the ribs and roof of Northern Coal Co.'s Rienaw No. 2 Mine in Colorado, with the intent of preventing spontaneous combustion after closing the mine. However, results were not reported.

The cited references indicate that sealants were generally individually and uniquely devised and prepared to fit a specific mine's conditions and needs. The choice of a sealant depended on various factors such as cost-effectiveness, availability of materials, and ease of preparation, in addition to its suitability to the need at hand.

The U.S. Bureau of Mines (USBM) examined several materials for sealing specific areas in a heaving mine floor, such as near stoppings and in longwall entries. Such sealants should be elastic in nature, have a long shelf life of at least a few months, or for the period needed to afford safe mining, and be stable in a mine atmosphere. In addition, such sealants should be easy to prepare and cost-effective. These sealants would also be effective in mines with a spontaneous heating problem, but without floor heave.

Three sealant systems were chosen for study. They were bentonite-water mixtures, sodium silicate-based gels, and guar gum-based gels. The work done with these sealants is described in the following sections.

SEALANTS

BENTONITE-WATER MIXTURES

The bentonite used in the tests, Volclay bentonite PLS-50, was obtained from American Colloid Co. This sodium bentonite is chemically and polymerically treated. In its final form it is free-flowing and has a high swelling potential with water. It swells up to 13.8 times its volume in water at full saturation, and in the process it absorbs nearly five times its weight in water. On drying it shrinks to its original volume. Swelling is reversible and can be done an infinite number of times, if the water used is fairly pure. When mixed with 7 to 10 parts water, gelatinous or creamlike pastes form. The bentonite can be mixed with water with or without mechanical devices, such as pumps or mixing paddles. When mixing devices are not used, the Volclay bentonite is added slowly to a measured quantity of water and allowed to swell and form a gel. However, clay-bentonite water mixtures are not gels in the strict sense of the word. They are suspensions of very fine particles but do not possess the molecular bonds that form in proper gel structures.

Mixtures of bentonite with water, varying in concentration from 4 to 8 pct of bentonite, were prepared and placed in either empty glass trays or were poured into glass trays lined with coal lumps or with coal lumps covered with thin layers of rock dust. The glass trays were left open to the ambient atmosphere. All the mixtures in the trays dried and cracks formed in them within a few days. Rewetting the dried bentonite layers by spraying them with water or by adding water to the trays, did not result in renewed gels or creamy pastes of the original quality. Distilled water was used in all the preparation steps. However, electrolytes must have leached from the coal and rock dust into the water, and these dissolved electrolytes affected the ability of the bentonite to form gels. In the presence of electrolytes the bentonite clay forms flocs, which settle. The higher the valency of the cations of the electrolytes, the stronger the effect. Repeated tests with various concentrations of bentonite gave similar results. To slow down the vaporization of the water, hydrated calcium chloride ($CaCl_2 \cdot XH_2O$) flakes were sprinkled on top of freshly prepared bentonite layers, after the layers were poured on the coal pieces in the trays. The flakes dissolved in the paste, but probably remained concentrated at the surface portions of the layers. Calcium chloride is a highly hygroscopic salt and has been used for the sorption of water vapor from air (21). The procedure did slow down the dehydration process, but did not prevent it. Based on these initial results, no additional tests were conducted with the bentonite mixtures.

SILICATE GELS

A technical sodium silicate solution, or water glass [$Na_2SiO_3 \cdot (H_2O)_n$], of 40° to 42° Bé[3] was used in the preparation of the gels.

Aqueous solutions of the alkali silicates hydrolyze extensively, as follows:

$$2Na_2SiO_3 + H_2O \rightarrow Na_2Si_2O_5 + 2NaOH.$$
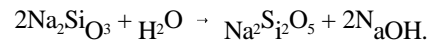
If the silicate solutions are acidified, mononuclear orthosilicic acid [$Si(OH)_4$] is liberated. It exists in equilibrium with its conjugate bases, $SiO(OH)_3^{-1}$ and $SiO_2(OH)_2^{-2}$ (22). With time, the silicic acid condenses to polymeric aggregates of polysilicic acid, as follows (23):

$$\geq Si\text{-}OH + HO\text{-}Si \leq \rightarrow \geq Si\text{-}O\text{-}Si \leq + H_2O.$$
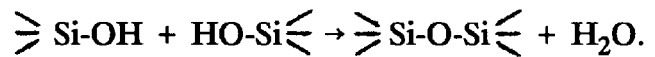
Initially, the aggregates remain dispersed as a colloid, but finally they set to a gel, with complete immobilization of the fluid. The addition of electrolytes, provided the solution is not too dilute, accelerates the gelling time, which depends on the hydrogen ion concentration (pH) of the mixture. Silica gels rich in water are transparent, soft, and fairly elastic. Freshly prepared gels may contain over 200 moles of water per mole of SiO_2 (24).

Various electrolytes were evaluated as gelling agents to accelerate the gelling time. They included ammonium sulfate [$(NH_4)_2SO_4$], ammonium chloride (NH_4Cl), monoammonium orthophosphate ($NH_4H_2PO_4$; MAP), sodium tetraborate decahydrate ($Na_2B_4O_7 \cdot 10H_2O$; borax), sodium nitrate ($NaNO_3$), and calcium chloride dihydrate ($CaCl_2 \cdot 2H_2O$), among others. Concentrations of ingredients used in the preparation of silica gels are shown in table 1. Good gels that were soft, relatively elastic, and structurally stable formed with ammonium sulfate, borax, and MAP. With some of the other salts either slurries formed, which rapidly precipitated, or the solutions remained transparent and fluid. Gelation time depended on the relative proportions of the various ingredients and varied from 1 to about 3 min. The gels prepared with the borax took much more time to gel. When small amounts of ethylene glycol (CH_2OHCH_2OH) were used as an additional solvent, gelation times were between 10 and 15 min. Another

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[3]Baum′e (B′e) gravity scale for liquids heavier than water is defined as follows: Degree Baum′e = $145 - \dfrac{145}{G}$.

A specific gravity, G, of 1 at 60 °F (16 °C) [relative to the specific gravity of water at 60 °F (16 °C) corresponds to 0.0° B′e.

Table 1.—Experimental silica gel formulations

Electrolyte	Electrolyte weight, g	Water, mL	Water glass, mL	Gelation time, s	Comments
$(NH_4)_2SO_4$	1.0	23	5.0	~40	No mixing.
$(NH_4)_2SO_4$	1.0	25	3-4	60	None.
$(NH_4)_2SO_4$	1.0	26	3.0	65	Do.
NH_4NO_3	1.0	27	3.0	60	Ammonia smell.
NH_4NO_3	1.3	27	3.0	50	Do.
NH_4Cl	1.0	25	3.0	NAp	Slurry formed; ammonia smell.
NH_4Cl	1.4	27	3.3	NAp	Do.
$NH_4H_2PO_4$	1.0	25	3.0	20	None.
$NH_4H_2PO_4$	1.0	27	2.7-3.0	130	Do.
$NH_4H_2PO_4$	1.0	30	2.5	160	Soft gel.
$NH_4H_2PO_4$	1.0	30	3.0	180	Do.
$NH_4H_2PO_4$	1.0	26	3.3	NA	Mixed slowly; gelled slowly.
$NH_4H_2PO_4$	1.3	27	3.3	110	None.
$Na_3PO_4 \cdot 12H_2O$	1.0	25	3.0	NAp	No gelling; pH = 12.
$Na_3PO_4 \cdot 12H_2O$	1.3	27	3.3	NAp	Do.
$NaNO_3$	1.0	25	3.0	NAp	No gelling.
$CaCl_2 \cdot 2H_2O$	1.0	25	3.0	NAp	Slurry formed; pH = 8-9.
$CaCl_2 \cdot 2H_2O$	0.5	25	3.0	NAp	Do.
$Na_2B_4O_7 \cdot 10H_2O$	1.0	25	3.3	NA	Slow gelling; pH = 10.
$Na_2B_4O_7 \cdot 10H_2O$	1.4	27	3.3	1,500	None.
$Na_2B_4O_7 \cdot 10H_2O$	1.3	29	3.3	3,600	Do.
$Na_2B_4O_7 \cdot 10H_2O$	1.0	27	4.0	Nap	No gelling.
$Na_2B_4O_7 \cdot 10H_2O + NH_4H_2PO_4$	0.5 / 0.8	27	3.3	NAp	Slurry formed; ammonia smell.

Do.   Same as above.
NA   Not available.
NAp   Not applicable.

alcohol methanol (CH_3OH), has been gelation times of certain silicic acid solutions by 50 pct or more (*25*).

With time, the gels lost water, shrank, and eventually dried out and crumbled into a powder. Some gels were kept in a humidity chamber, but these also lost water, although at a slower rate than the gels that were kept open in the laboratory air. The partially dried gels and the more extensively dried gels did not retain good structural cohesion and were not deemed suitable for sealing purposes in a mine.

The tendency for slowly drying bodies to crack has been noted for clay (*26*) and for silica gels (*27*). A microscopic observation of the dehydration process undergone by samples of sodium silicate gels (*28*) has shown that, after an induction period, cracks appear at random sites and that subsequent cracks originate on the surfaces of the initial cracks. This process is followed by the formation of aggregates of cracks, which then coalesce and eventually cover the entire surface. Finally, this dehydration front moves into the sample.

Silica and alumina gels are used in industry as precursors in the preparation of glass by pyrolysis of the gels. Often,

shrinkage occurs and cracks form in the specimens during the drying stage. Various drying procedures have been devised to eliminate cracks and shrinkage; however, good results are not always obtained. For use as sealants in a mine, extensive drying, shrinking, or excessive cracking is not tolerable.

Plain water glass will dry in air into a transparent hard glass. It will retain some of the water, with the amount depending on the thickness of the layer formed (*29*). Chunks of coal were coated with the water glass and left to dry in an open tray in the laboratory. In about a week, a transparent hard layer of glass formed on the coal pieces and completely sealed them. The glass layers retained their integrity. Another property of silicates that is of interest to a mine that contains reactive pyrites, is the ability of the silicate to form insoluble products with ferrous and ferric ions. Such insoluble silicates will retard the oxidation of the pyrite. Thus, the application of plain water glass in a coal mine that contains pyrites merits additional attention. Another attractive feature of inorganic silicates is their nontoxic and fire-resistant nature. Drying time of the water glass is long. While drying, it can seep into the ground

and away from the area where it is needed. Special application procedures will have to be developed to contain the solution.

GUAR GUM GELS

Various materials, both natural and manmade, can form gels. These gels consist of polymers immersed in a liquid medium. The liquid prevents the polymer network from collapsing while the network holds the liquid in place. Cellulose compounds, starches, saccharides (sugars), and proteins are among the natural gel-formers, whereas a polymer such as polyacrylamide is an example of a manmade gel-former.

The gel chosen for this study as a sealant was guar gum. Guar gum belongs to a group of hemicellulose compounds (sugars that contain cis-1,2-diols such as mannose and galactose) that include substances found in fruits and in the endosperm of seeds of various plants, such as quince, flax, and psyllium, and in locust bean and locust kernel. Guar gum is a complex carbohydrate polymer of galactose and mannose (30), or galactomannan. A monomer unit is as follows: although these galactomannans can differ widely in their
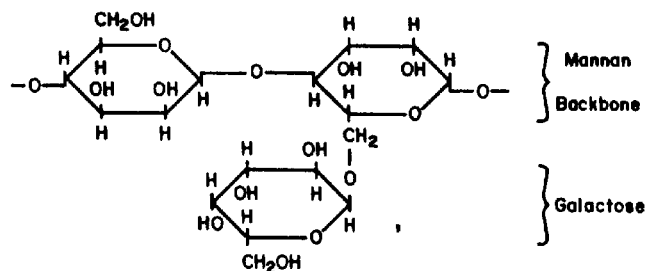
galactos/mannose compsition ratios. Guar gum has many uses in the food, paper, cosmetic, and pharmaceutical industries, as well as in mining and in the production of explosives (31). Many derivatives of guar gum are available commercially. Hydroxyalkylated gums are soluble in water-miscible solvents such as glycols, and are used in the production of various explosives. Other hydroxyalkylated gums are compatible with calcium salt solutions and are used in the preparation of aqueous slurry explosives and in oil well fracturing.

Two guar gums, Jaguar 2332 and Jaguar 2379, were obtained[4] for initial tests. These two are both self-crosslinking blends of guar with a crosslinker and pH buffers, and were chosen for ease of preparation.

PREPARATION METHODS OF GUAR GUM GELS

ELECTROLYTES

Initial mixtures of the two guar gums with water only, produced viscous liquids that did not gel. Electrolytes were required to change the pH of the solutions so that they would gel. Electrolytes tested were sodium chloride (NaCl), lithium chloride (LiCl), potassium chloride (KCl), $CaCl_2 \cdot XH_2O$, barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$), borax, sodium bicarbonate ($NaHCO_3$), MAP, and calcium sulfate ($CaSO_4$). Good gels formed with hydrated calcium chloride, with MAP, or with a mixture of the two.

INGREDIENT PROPORTIONS AND AMBIENT CONDITIONS

Mixtures were prepared at various ratios of guar gum, electrolyte, and water in order to identify optimal mixtures. Properties observed were gelation times, weight loss, syneresis (expulsion of liquid from the gel network due to induced contraction, brought about by additional bond formation after initial gelling, or by breakdown of the gel), and shrinkage. Adhesion to the walls of the containers was also checked at intervals.

The effects of high relative humidity on the properties of these gels were monitored by placing duplicate samples of the gels in high-humidity chambers.

INHIBITION OF BACTERIA AND MOLD FORMATION

Guar gums are sugars and, therefore, are easily attacked by bacteria and mold. The presence of electrolytes does not deter or prevent spoilage. Small bacteria/mold colonies of varied colors were observed on the surfaces of the gels within a few days or within a week or two of preparation time. If left undisturbed, the colonies grew in size; with time they affected the gel structure and eventually destroyed it.

Three inhibitors were evaluated for the gels. They were sodium benzoate ($NaC_7H_5O_2$), a preservative, and two commercial preparations (recommended by the producers of the guar gum), Dowicil 75[5] and Ottasept.[6] Of the three compounds, only Ottasept prevented formation of colonies of any kind, over periods of time as long as a year, and was used in all subsequent gel preparations.

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[4]Hi-Tek Polymers, Inc., Jefferson, KY. Now Rhone-Poulenc, Louisville, KY 40299.

[5]Dowicil 75; Dow Chemical USA, Western Division, Pittsburg, CA 94565.

[6]Ottasept technical; Ferro Corp., Ferro Chemicals Group, Bedford Chemical Division, Bedford, OH 44145-4494. Ottasept has since been sold to Nipa Hardwicke, Inc., Wilmington, DE, and is now sold under the trade name Nipacide-PXR.

OPTIMAL GEL MIXTURES

Two formulations gave overall satisfactory results. Thereafter, these were used in small laboratory-size preparations as well as in batch preparations for both laboratory and mine applications. These formulations are shown in table 2.

Table 2.—Guar gum gel formulations

(weight in g)

Ingredients	Formulation 1	Formulation 2
Water	1,000	950
Ethylene glycol	—	50
Jaguar 2379	23	23
$CaCl_2 \cdot 2H_2O$[1]	20	20
Ottasept technical (Nipacide-PXR) .	2	2

[1]When $CaCl_2 \cdot H_2O$ is used in the preparation, the weight is modified accordingly.

Both formulations resulted in stable gels, which lasted for long periods of time. Ethylene glycol was used in formulation 2 to improve gel strength. Sugars and polyols are known to modify gelation properties of gelatin gels (*32*) and of polysaccharide gels such as carrageenan (*33*), and to increase gel strength in each case. Carrageenan is similar to guar gum; it is characterized by an alternating disaccharide unit of two types of galactose. Thus, it is highly probable that the same effect of increasing gel strength and improving quality, will be noted for guar gum gels, as well.

It is important to note that both formulations consist mostly of water. The concentration of water is 96 and 91 pct by weight in the two formulations, respectively. In case of a heating nearby, they can also act as coolants, to quench or otherwise extinguish a self-heating. In addition, the $CaCl_2$ salt can act as an adsorbent for water present as moisture in the mine air, and aid the quenching. Calcium chloride is used in some underground coal mines as an inhibitor for self-heatings; thus, it can also act in that capacity. As for the guar gum, it is present in small amounts, and although it is a fuel, will not contribute much to the fuel load.

EFFECTS OF COAL, ROCK DUST, AND MINE FLOOR MATERIALS

Once formulations 1 and 2 were selected, their performance when in contact with coal, rock dust, or other materials likely to be found in mine floor strata, was of paramount importance. Thus, gel mixtures were prepared and poured into trays and larger containers lined with coal pieces and covered with rock dust, and into trays and larger containers lined with various rock material and/or clay, to simulate mine strata. These gels were observed for periods as long as 2 to 3 months. When the gels contained Ottasept they performed well, and were not affected by the coal, rock dust, or other materials. That is to say, they retained all their desirable characteristics of elasticity, stability and adherence to the walls. Figure 1 shows a freshly prepared gel being poured into a large glass jar containing rocks and rock dust; figure 2 shows the appearance of the gel after 1 week in the jar.

MIXING PROCEDURES

When water was the only solvent, the dry ingredients were weighed separately and then mixed together in a container. Water was then added to the dry powder at a fast rate while mixing with a glass rod when small quantities were prepared in a beaker, or with a wooden mixing paddle when larger quantities were prepared. Mixing continued until all the powder was dispersed in the water, and the gel started to form. By that time, the powder usually had dissolved.

When both water and ethylene glycol were used, the dry powders were weighed and mixed together in the same way. Ethylene glycol was then added to the powder mix, and the powder was dispersed in the small amount of ethylene glycol by mixing. Water was then added to the glycol-powder combination. This procedure was helpful in the preparation of the larger batches for the mine. For these latter tests, the glycol-powder combination was prepared in the laboratory and then taken to the mine. The addition of water and the final mixing was conducted in the mine.

BATCH SCALE AND MINE TESTS

GELS IN TUBS

The next step was to observe the performance of the gels in a mine environment. Large plastic tubs (76- by 61- by 15-cm deep) were placed in selected areas of the USBM Safety Research Coal Mine, so that the gels would be exposed to various ventilation flows and mine conditions. The tubs were lined with coal chunks from the mine (Pittsburgh Coalbed), and rock dust was sprinkled on top of the coal chunks.

Initially, the preparation of large batches, 30 L each, was practiced in the laboratory. The rate of addition of water to the glycol-powder mixture, and the rate of mixing was important for obtaining good gels. Both wooden paddles and a handheld electric mixer were evaluated. Better gels were obtained with the wooden paddle. When

the preparation procedure was established, gels were prepared in the mine and poured into the tubs. Figures 3 to 6 depict some of the steps in the preparation of the gels in the mine. Four tubs were filled with gel and the gel was observed for a year. During this time, temperatures as low as 4 °C and as high as 21 °C were recorded; likewise, relative humidities ranged from 15 to 100 pct, but they predominated on the high side. Low relative humidities were noted only during late October and early November 1991. The high relative humidity slowed the evaporation of the water from the gels and was beneficial to gel stability.

During the test period, the gels lost some water and their height in the tubs was lowered by about 2 to 3 cm. However, throughout that time they continued to adhere to the sides of the tubs, and bacterial colonies did not form. Mine dust, both coal and rock dust, settled on the surface of the gels in the tubs and may have helped to retard the loss of water to a small extent. Within 3 or 4 months, the surface layers of the gel also changed texture. A thickened and somewhat crinkled skin of gel that included the accumulated dust, formed on top.

GELS IN THE MINE FLOOR

While the gels were aging in the tubs, three pits were dug in the mine floor, two in one area in May 1991, and one in another area in July 1991. During that time of year, both relative humidity and temperature in the mine were relatively high. The size of the first two pits was 91- by 46- by 23-cm deep; the third pit was 76- by 38- by 15-cm deep. All of the pits had slightly sloping sides. Coal pieces and some rock dust lined the bottom of the third pit. Gel batches of 30 L each were prepared using the formulation with ethylene glycol. Weighing and mixing of the dry powders were done in the laboratory. Mixing with the fluids was done in the mine at the site of the pits. These gels were observed for a year. They, like the other gels in the laboratory or the tubs, lost water over time and receded to a lower level by about 2 to 3 cm. However, throughout this period, the gels adhered to the side walls and appeared to form a good seal, as seen in figures 7 and 8.

CONTINUOUS MIXING PREPARATION

The batch preparation of gels is easy and fast. For sealing small areas, such as near stoppings, this is a satisfactory method. For sealing larger areas in a mine, a method for continuous mixing of the ingredients is required. Two methods were appraised for the continuous preparation and application of gels in the mine: (1) the use of an eductor for mixing the powder with water and (2) the use of a gunite machine for mixing and application purposes.

EDUCTORS

An eductor is a device that utilizes the cavitation produced at a pipe constriction, or venturi, through which water is pumped at a high velocity, for mixing purposes. When a powder is fed into the water stream, it will be carried by the stream out of the eductor, and at the same time will be mixed with the water, especially if it is water-soluble. Both small and large eductors were used in the tests. The eductors were equipped with a variety of components, such as a funnel to feed the powder, piping to hold a static mixer at the output of the eductor, and a hose to direct the flow of the final mixture to the desired location. Valving was also utilized to control the rate of water flow. Figures 9 and 10 show the arrangements of the small and large eductors, respectively.

The amounts of guar gum, calcium chloride, and inhibitor needed per unit time for a specific water flow rate, were calculated, and a mixture of the powders was then prepared to suffice for a chosen time period of water flow. This mixture of the powders was manually fed through the funnel into the water stream, and the resultant gel was directed into various containers, placed in the mine, for observation.

Initial tests indicated that neither the granular nor the flake-type calcium chloride that are available commercially, dissolved or mixed well with the water. The granular calcium chloride was then ground to a finer size prior to being mixed with the guar gum. This powder mixture did mix well with the water and formed relatively good gels.

The use of static mixing elements to process viscous fluids is quite common. These mixing units contain intersecting channels with frequent change of direction. They accelerate mass transfer and produce dispersions due in part to their large interfacial area. Pressure drop in these units is low. Static mixing elements are available in different sizes and designs; a few of these were used in the flow tests with the eductors. The static mixing elements with a more open design did not get clogged and assisted in the mixing, while the ones with a more convoluted design, or twist ratio, did get clogged with gel.

This method of continuous mixing and application of a gel appears feasible. Additional testing is required to refine the design. Bins or containers to hold the dry ingredients, in conjunction with a feeding screw, or two feeding screws, one for guar plus inhibitor and one for calcium chloride, are needed for a more automated mixing process.

GUNITE MACHINE

A gunite machine is used to pneumatically apply a Portland cement mortar to various areas in the mine. The cement and sand are mixed, wetted with water, and applied to roof, roads, ribs, and other mine areas as a sealing agent to prevent deterioration. Since the gunite machine is present in many mines and essentially performs the same steps that are required to prepare a gel, it was tested as a means for gel preparation and application. A small gunite machine at the USBM's Lake Lynn Laboratory near Fairchance, PA, was cleaned and used. Results were not satisfactory. A specified ratio of water to solids is required to form a gel; this ratio could not be obtained with the gunite machine. In addition, the mixing mode of the water stream with the mixed powder of guar gum and calcium chloride was not conducive to gel formation. It is possible that the gunite machine could be modified to permit the desired ratio of water to solids and to afford the needed mixing mode.

COMMERCIAL MIXING UNITS

Polymeric flocculating agents are used in water or waste water treatment plants to enhance settling, to aid filtration or to accelerate solids dewatering. These polymers are available in three basic types: dry powder or granules, emulsion, and neat solution. Equipment for wetting, diluting, mixing, and blending the different types of polymers with the water varies from the more simple to the

highly automatic. The choice of equipment depends on the type of operation, continuous or infrequent, the size of the operation, the degree of flexibility required, and the cost. Simplified systems, with some degree of manual feeding in a batch fashion, can reduce the cost, but can result in poor mixtures. Polymer feed systems, some of which could be applied to gel preparation, are described elsewhere (*34*). Some of these systems should be evaluated and may prove highly effective for the preparation and application of gels in a mine.

ADDITIONAL MODIFICATIONS

Slight modifications in gelling time can simplify continuous mixing procedures. The effects of temperature on gelling time were evaluated by using both cold and warm water in the preparation of the gels. Water temperatures as low as 10 °C and as high as 70 °C were utilized. Gelling time was shortened by the warm water and was slightly extended by the cold water. In either case, the change was less than a minute and was not enough to affect the gelling time significantly. Reducing the quantities of guar gum and/or $CaCl_2$ by very small amounts (1 to 3 pct) will increase the gelling time without affecting the overall characteristics and performance of the resultant gels. Longer gelling times will permit the use of longer mixing sections in the eductors. The resultant gels will have good consistency and will not clog the mixing elements. Changes in the pH values of the gels may also affect gelling times, and are worthy of consideration.

COST OF GEL

The 1995 cost of ingredients to make 100 gal (378.5 L) of guar gum gel is shown in table 3.

The guar gum gel is costly; both the guar gum and the inhibitor are expensive. However, if used selectively to prevent

heatings in specific areas, it would be cost-effective. The cost for a mine will depend on the area to be covered and the depth of the gel needed to seal each specific location.

Table 3.—Cost of ingredients to prepare guar gum gel

[Cost basis: 100 gal (378.5 L) gel]

Ingredients	Amount		Cost			Total cost, $
	lb	(kg)	$/lb	($/kg)	Based on—	
Guar gum, Jaguar 2379	19.2	(8.71)	1.10	(2.43)	50-lb bag	21.11
$CaCl_2 \cdot H_2O$ flakes	16.7	(7.58)	0.23	(0.51)	50-lb bag	3.84
Ottasept (Nipacide-PXR)	1.7	(0.77)	8.05	(17.75)	50-kg drum	13.44
Total cost ($) per 100 gal (378.5 L)						38.39

NOTE.—Calculations were made using U.S. customary units, which are standard in the industry.

SUMMARY AND RECOMMENDATIONS

A guar gum-based gel was developed for use as a sealant in coalmines that undergo self-heatings, and especially in mines with both floor upheaval and self-heatings. The gel is easily prepared, in batch form, from a few ingredients. It is elastic in nature and has a useful life in excess of a year. The gel is to be used in susceptible areas in the mine to minimize air ingress and thereby prevent, or at least slow down, self-heatings. For mines that undergo self-heatings and minimal upheaval, only small, specific areas will require elastic sealants. These mines can prepare the gel in batch mode, mix them manually, and pour directly from the container onto the areas that require sealing. The dry ingredients for a batch can be preweighted and kept in plastic bags, such as "zip-lock"

bags. The guar gum and inhibitor can be kept together in one bag; the calcium chloride flakes or granules can be stored in another bag. For immediate use, all three ingredients can be placed in one bag. The preparation of the gel in a continuous mode requires additional development. The use of the eductors for mixing and applying the gels is feasible, but needs additional modifications. The use of commercial units is even more attractive. A variety of mixing units that will be able to prepare and apply the gels are available. The optimal ones will have to be found by trial. The gel is costly and is recommended for use as a sealant only in specific areas in which rigid sealants would not suffice due to cracking.

ACKNOWLEDGMENTS

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Figure 1.—Freshly prepared gel being poured into a jar containing rocks and coal dust.

Figure 2.—Appearance of gel after 1 week in a jar.

Figure 3.—Two plastic tubs with coal pieces and rock dust are being placed next to the coal rib while dry ingredients with ehtylene glycol are being mixed prior to the addition of water.

Figure 4.—Mixing of all the ingredients with a wooden paddle to form a gel.
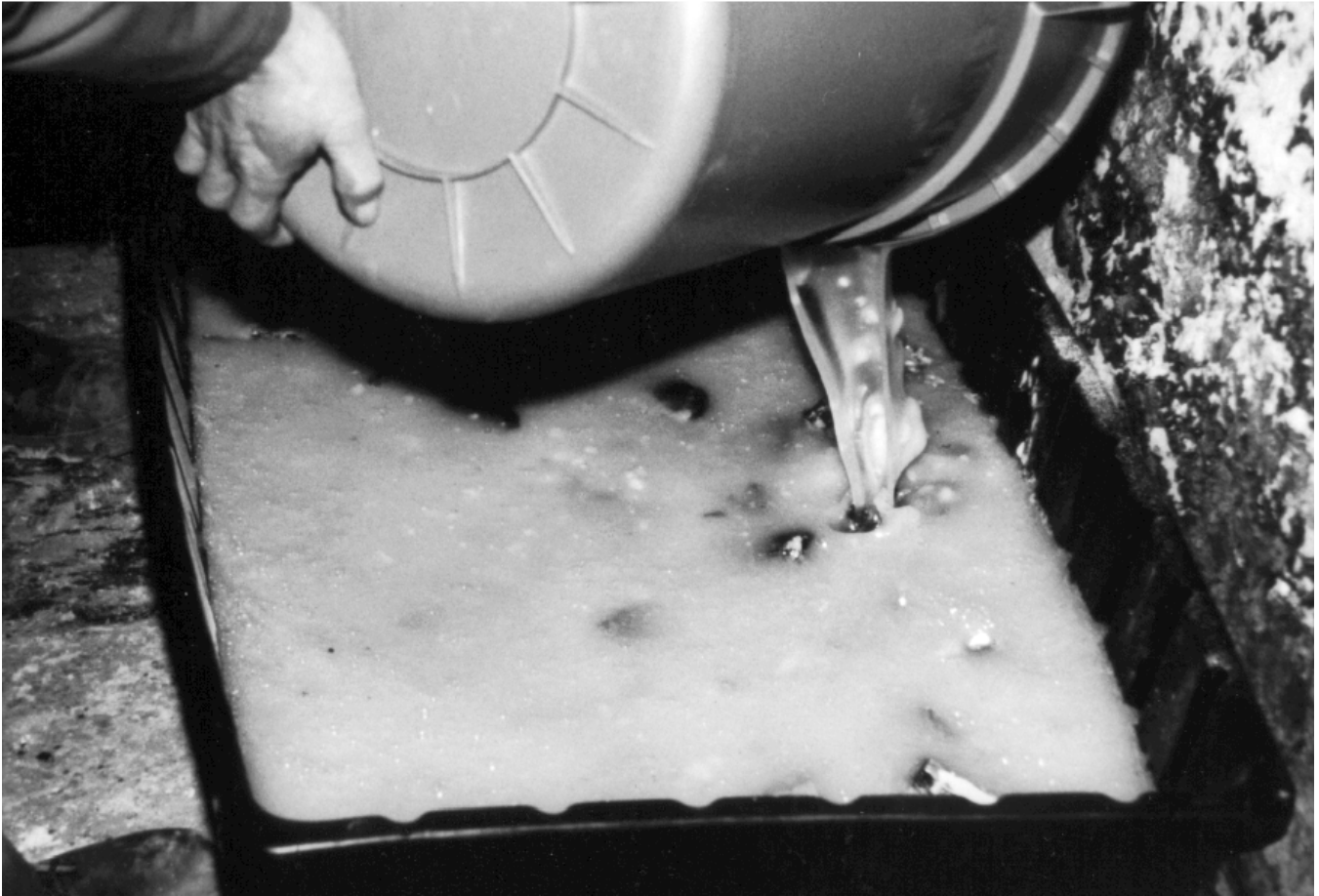
Figure 5.—Freshly prepared gel is poured into a tub.

Figure 6.—Gel in tub in underground mine.

Figure 7.—Freshly prepared gel in pit in mine floor.

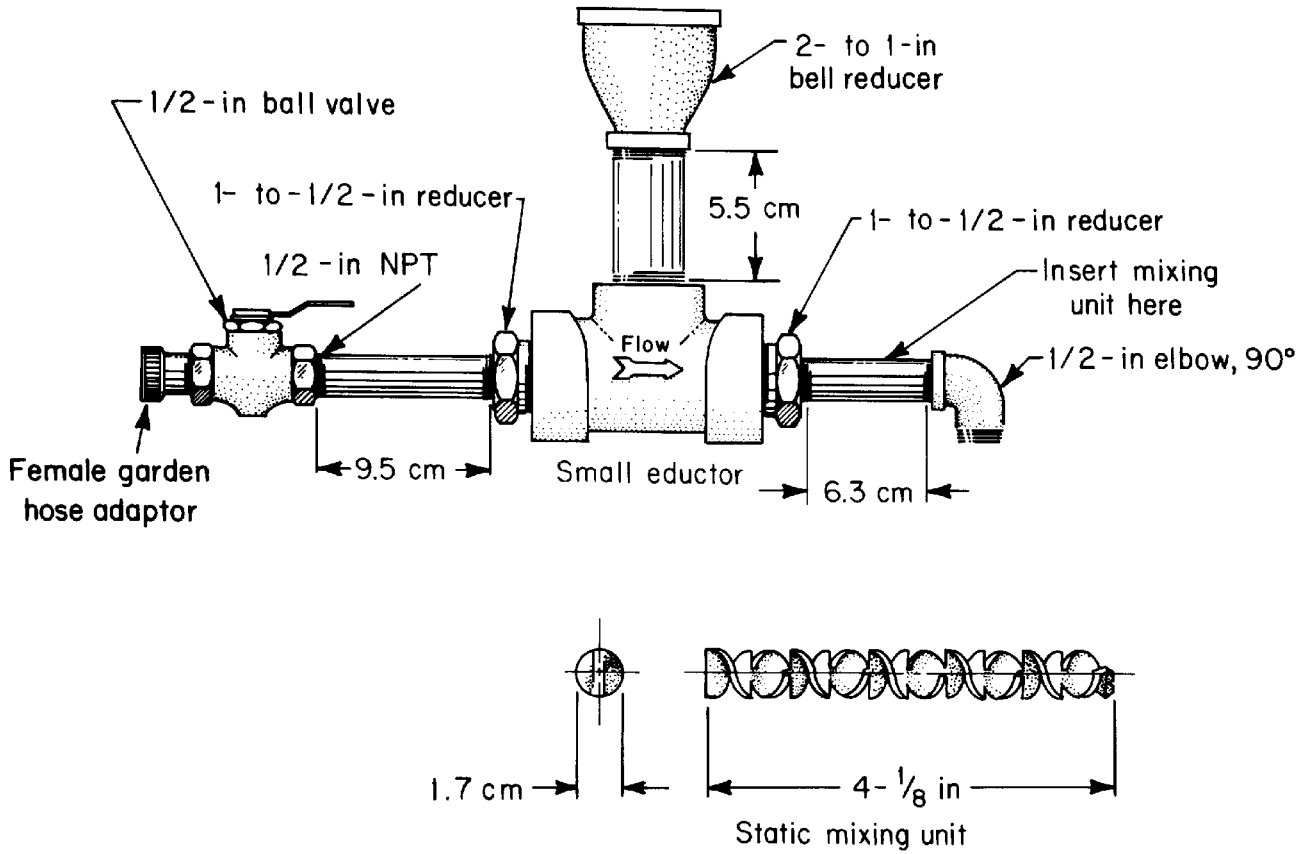**Figure 8.—Appearance of the gel after 3 months in pit in mine floor.**

1/2-in ball valve

1- to-1/2-in reducer

1/2-in NPT

2- to 1-in bell reducer

5.5 cm

1- to-1/2-in reducer

Insert mixing unit here

Flow

1/2-in elbow, 90°

Female garden hose adaptor

9.5 cm

Small eductor

6.3 cm

1.7 cm

4-1/8 in

Static mixing unit

Figure 9.—Small eductor arrangement.

1-1/2- to 3/4-in reducer

1-1/2-in NPT

1-in NPT

1- to 1/2-in reducer

1/2-in ball valve

Flow

Insert mixing unit here
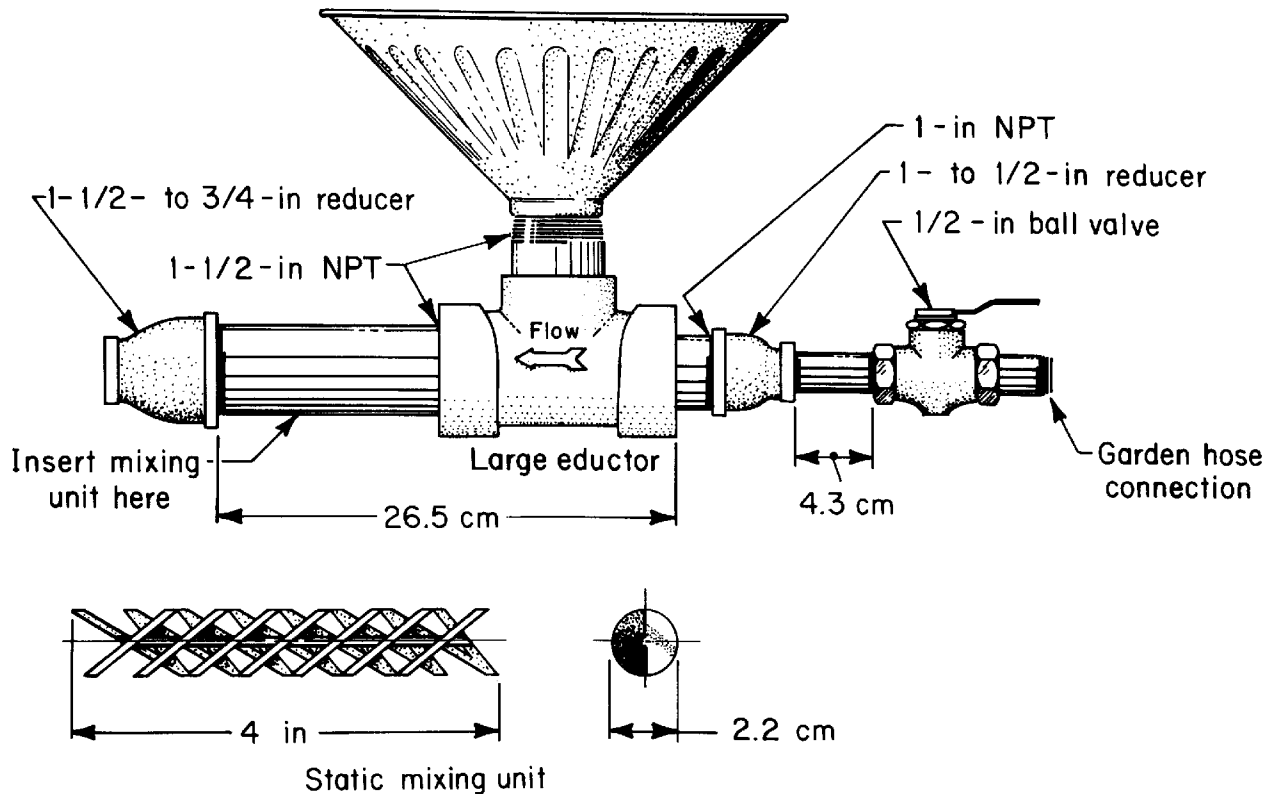
Large eductor

26.5 cm

4.3 cm

Garden hose connection

4 in

2.2 cm

Static mixing unit

Figure 10.— Large eductor arrangement.