United States Patent [19]

Slobod et al.

[11] **3,845,632**

[45] **Nov. 5, 1974**

[54] **METHOD OF SEALING COAL AGAINST METHANE EMISSION**

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[22] Filed: **May 21, 1973**

[21] Appl. No.: **362,280**

[52] **U.S. Cl.** ..... **61/36 R,** 299/12

[51] **Int. Cl.** **E02d 3/14,** E21f 15/00

[58] **Field of Search** ..... 61/36 R; 299/12; 102/22, 102/23

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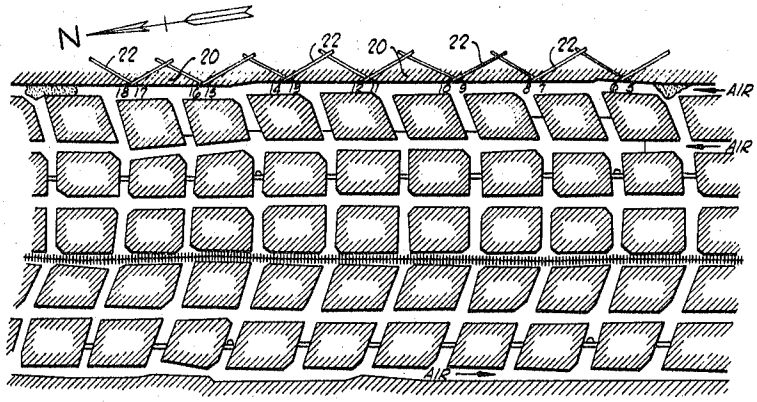
"Degasifying Before Mining" William M. Merrits, p. 74–78, Coal Age, August 1961.

Primary Examiner—W. C. Reynolds
Assistant Examiner—Alex Grosz
Attorney, Agent, or Firm—Dunlap, Laney, Hessin, Dougherty & Codding

[57] **ABSTRACT**

A method of sealing cracks in a coal seam to prevent methane gas from passing out of the seam at a face thereof comprising injecting a gelable, pumpable silicic acid composition into the cracks in the coal, and allowing the silicic acid composition to set to a gel forming a barrier against gas migration.

16 Claims, 5 Drawing Figures



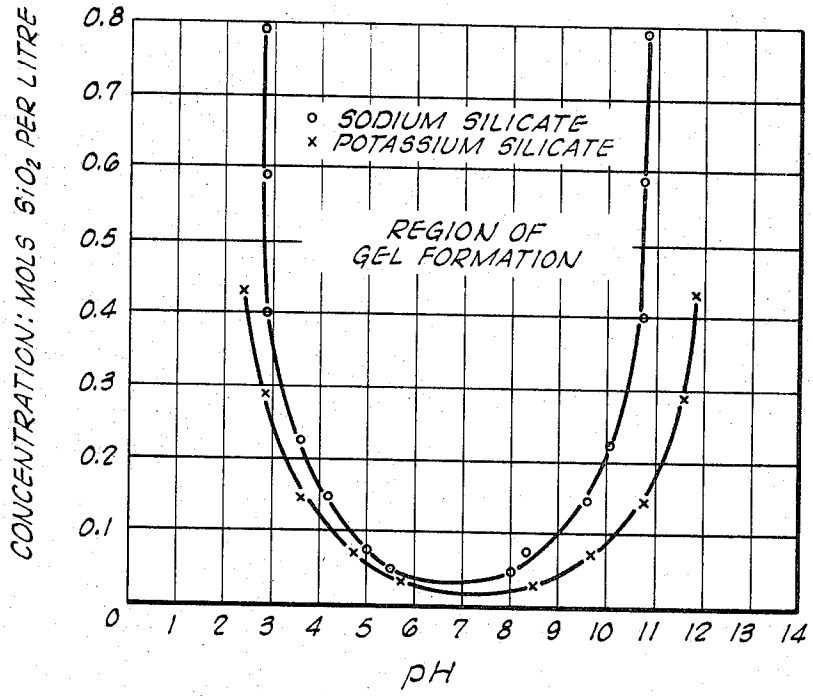
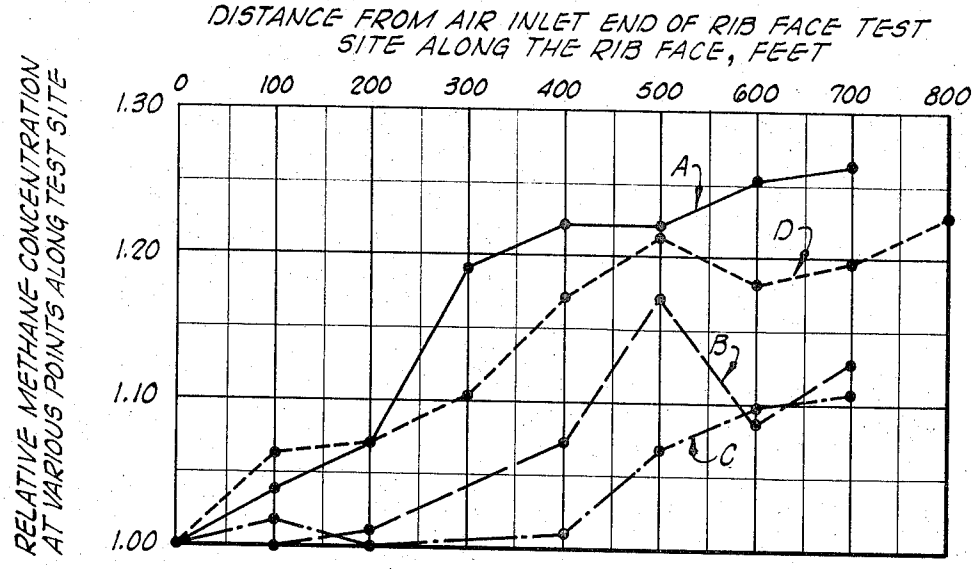


FIG. 1



A. TESTS DECEMBER 8, 1971 PRIOR TO INFUSION
B. TESTS DECEMBER 21-22, 1971 AFTER 300 FEET OF RIB FACE INFUSED
C. TESTS DECEMBER 29-30, 1971 AFTER 500 FEET OF RIB FACE INFUSED
D. TESTS JANUARY 11, 1972 AFTER 700 FEET OF RIB FACE INFUSED

FIG. 4

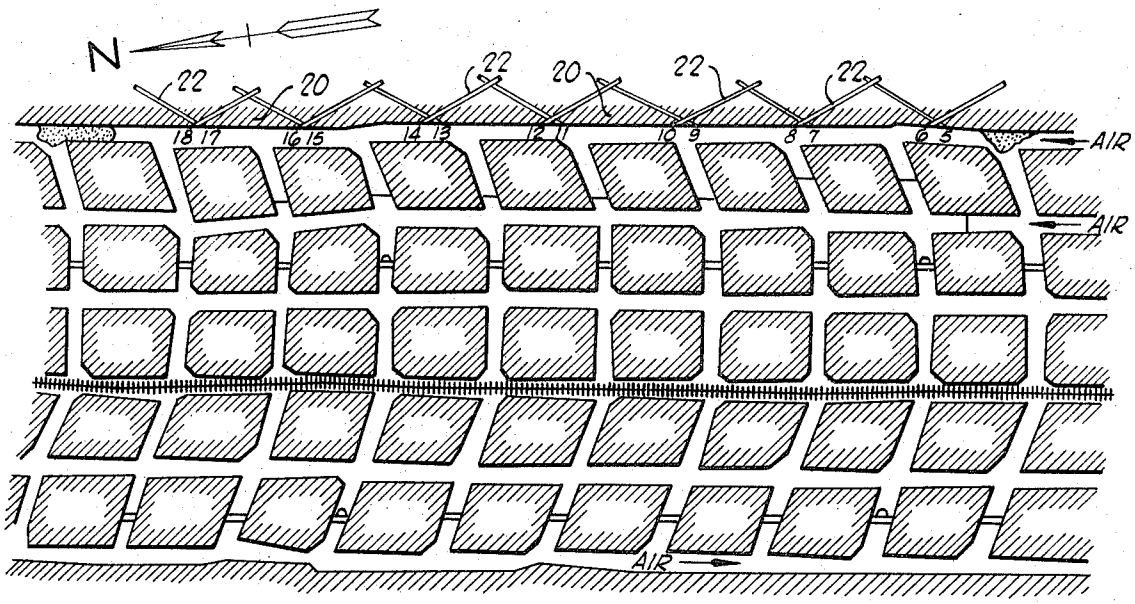

N
22
20
22
20
22
22
18 17
16 15
14 13
12 11
10 9
8 7
6 5
AIR
AIR
AIR

FIG. 2

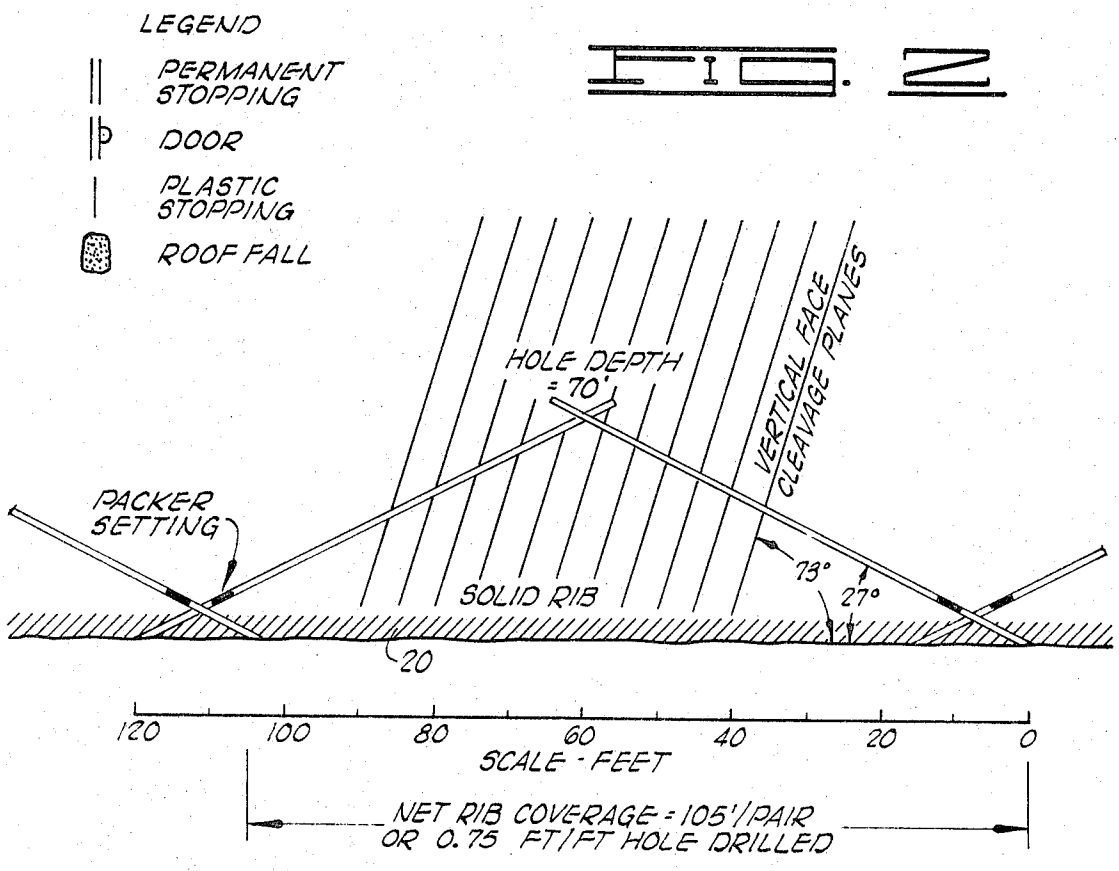
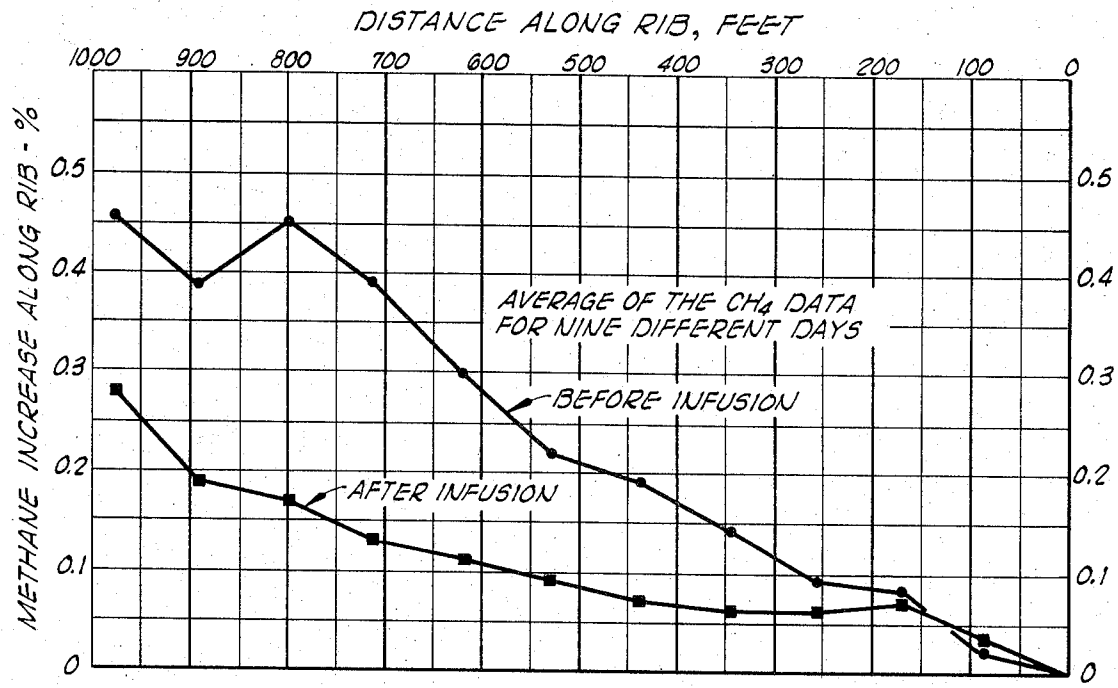

LEGEND
PERMANENT STOPPING
DOOR
PLASTIC STOPPING
ROOF FALL
HOLE DEPTH = 70'
VERTICAL FACE CLEAVAGE PLANES
PACKER SETTING
SOLID RIB
73°
27°
20
120
100
80
60
40
20
0
SCALE - FEET
NET RIB COVERAGE = 105'/PAIR
OR 0.75 FT/FT HOLE DRILLED

FIG. 3


DISTANCE ALONG RIB, FEET
1000
900
800
700
600
500
400
300
200
100
0
METHANE INCREASE ALONG RIB - %
0.5
0.4
0.3
0.2
0.1
0
AVERAGE OF THE CH_4 DATA FOR NINE DIFFERENT DAYS
BEFORE INFUSION
AFTER INFUSION

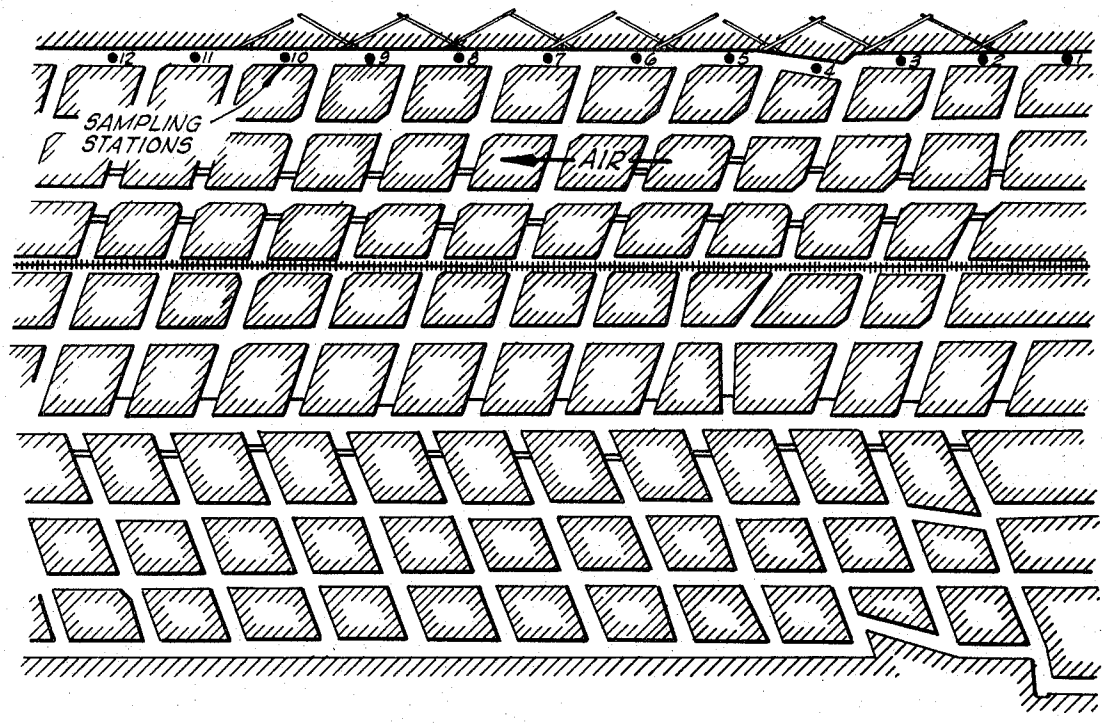

12
11
10
9
8
7
6
5
4
3
2
1
SAMPLING STATIONS
AIR

FIG. 5

METHOD OF SEALING COAL AGAINST METHANE EMISSION

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention generally relates to coal production, and more particularly, but not by way of limitation, to a method of increasing the safety with which coal can be produced by preventing dangerous, excessive accumulations of methane gas at working locations in a subterranean coal mine.

2. Brief Description of the Prior Art

A serious hazard to safety and the health of workmen engaged in the mining of coal is contamination of the working environment with methane gas. Air circulating systems are used in subterranean coal mining to continuously supply fresh air to the working areas, and to remove stale and contaminated air from such areas. Two of the major contaminants which seriously and dangerously pollute the air adjacent the working face in mines are methane gas and coal dust. Methane gas is inherently present in varying degrees in the micropore structure of substantially all coals, and is present in significant quantities in the deeper, more gassy coals. Most bituminous coal seams are characterized in having bedding and cleavage planes extending through the seam and opening at the face of the seams, and these planes of cleavage are the locus of cracks or fissures of sufficient porosity that methane desorbed from the micropores in the coal can bleed or migrate through the channels thus formed to the outer surface of the seam. As a heading is developed in the coal seam during mining, these cracks are intersected as the mining face or rib faces cut across the planes of cleavage and bedding planes. The result is often a dangerous buildup of methane in the air adjacent the working face, or adjacent ribs at the sides of entries and returns. At times the accumulation of this noxious and explosive gas becomes so severe that mining must be terminated until the methane can be dissipated and its concentration in the air reduced.

Various proposals have been advanced for alleviating the safety hazard created by the gas emission from coal seams. In recent years, some attempts have been made to seal or block the cracks and gas flow channels in the coal by injecting water into the seam. This technique provided some deterrant to gas migration to a protected area of the rib or mining face, and was particularly useful in diverting some of the methane to locations other than that in which the water was injected, but the reduction in gas emission and the permanency of such reduction were less than optimum, and the water injection method has not been widely accepted.

Another procedure recently proposed has been the injection of foam yielding compositions into the seam. As the foam is generated in the cracks and fissures of the coal seam, the foam forms a barrier which tends to block or obstruct gas movement through the foam toward the surface of the seam. Unfortunately, the stability and strength of the foams thus far proposed have probably not been adequate to provide effective gas blockage over useful time periods.

In a different technology from that of coal mining — the production of oil and gas — the problem of migration of fluids in a subterranean environment is encountered in a different context. A problem frequently confronted is that of sealing off or blocking the flow of water through a sandstone or limestone formation into an oil or gas bearing strata. It is also sometimes desirable to plug or block certain relatively large fluid flow passageways within the drainage radius of a well bore in order to permit an acidizing or treating fluid to be pumped into the relatively small pores of the oil bearing strata for purposes of stimulating production.

In these procedures, various types of plugging or blocking materials have been used, and others have been proposed and tested, but not widely used. In some cases, the development of a solid precipitate in situ has served to block or obstruct the liquid flow to an adequate degree. On other occasions, the development of a high viscosity liquid, or a semisolid gel has provided the diverting or blocking function sought. Among a wide variety of compositions used in this way are silicic acid gels. The chemistry of these materials is well developed, and their use in oil and gas bearing sandstones for the described purpose is particularly recommended by their compatibility with the silicates and silicious minerals predominating in strata of this type. A number of United States patents have been issued on various processes and compositions in which silicic acid gels are employed for the purpose of plugging or obstructing the flow channels in an oil or gas bearing sandstone formation, and as typical of such patents may be cited U.S. Pat. Nos. 3,375,872; 2,208,766; 2,330,145; 2,198,120; 2,207,759 and 2,236,147.

BRIEF DESCRIPTION OF THE PRESENT INVENTION

The present invention is a new and effective method for reducing the emission of gas at an exposed surface of a coal seam. The method can be generally identified as that of implacing a silicic acid gel in the seam crack network at a location between the exposed surface to be protected and the gas accumulation within the seam.

Broadly defined, the method of the invention comprises the steps of drilling at least one hole into a coal seam in a direction to intersect a plurality of the planes of cleavage in the coal, then forcing a gelable silicic acid composition into the seam via such hole. The gelling time of the composition is selectively controlled to assure that gelation will occur very shortly after implacement in the desired blocking position in the coal, and not prior to such implacement.

The significant differences between the environment and conditions encountered in oil and gas production, and those characteristic of coal mining, dictate that the effectiveness of silicic acid gels as a gas blocking or diverting material in coal cannot be assumed, or even safely postulated without experimental verification. The porosity of sandstone is vastly different from that of coal. In the former, there is a continuous intercapillarity of very fine or small pores which exist between sand grains, and the capillary flow channels thus formed are tortuous, and multi-directional. A gel or other plugging or diverting material thus must occupy relatively small voids, and tends to be well anchored and immobilized because of the tortuosity of pores in which it is located.

In coal, the gas is first desorbed from the micropores and then bleeds from the interior of the seam to an exposed surface thereof through relatively large cracks or

fissures which generally coincide with the bedding planes and with the existing planes of cleavage. Other cracks developed by geological stresses experienced during formation of the seam may also provide gas flow channels to the exterior of the seam. Although the character and extent of development of such planes of cleavage and bedding planes vary in different types of coal and geological conditions of formation, they are generally present in bituminous coals. The cleavage planes encountered are normally of two types, referred to as face cleavage planes and butt cleavage planes. The gas flow channels which exist at these planes of cleavage are generally relatively large cracks as compared to the capillaries in sandstone (several orders of magnitude higher), and are generally monodirectional and extend for relatively great distances in the coal. Since the planes of cleavage are vertical, the cracks or fissures also have very large vertical dimensions. In summary, the porosity characteristic and crack geometry of coal indicate that the development of an effective gel barrier should be much more difficult in coal, than in oil and gas bearing formations.

A further portent of difficulty in gel sealing in coal is the character of the surface of the coal itself. Carbonacious materials are, of course, oleophilic (or hydrophobic) in character, and thus it may be anticipated that the interfacial bonding capability of a water based silicic acid gel, for example, will be much less in a coal seam than in a sandstone or limestone formation. Thus, displacement or distortion of the gel block would be predicted to more easily occur in coal as a result of "slumping," or simply through the exerted force of the impounded gas.

It is also a significant difference between the conditions encountered in oil and gas production and coal production that the difference in the depth at which the producing formation or seam is located gives rise to widely varying pressures at the working levels. Thus, the overburden pressures encountered in many coal mines is sufficiently low, when coupled with the relatively friable character of the coal, to impose a limit on pump pressures which can be used to inject the gelable material into the seam without concern for fracturing the seam, and perhaps thereby further aggravating the problem of gas infusion. This limitation in turn limits the viscosity of the gelable material which can be initially made up and effectively pumped into the seam. Due to the much greater depth at which oil and gas bearing strata are generally found, and the high pressures concomitantly encountered, higher pump pressures and higher viscosity materials can generally be employed without concern for undesirable disruption of the formation geometry.

Finally, as contrasted with oil and gas, in coal production it may be said that it is the producing formation itself which is mined and which constitutes the final product. Unlike petroleum, the coal is subjected to relatively little refining and virtually no change of chemical state in the course of such refining. Any gas blocking material, such as a gel, which is used in the seam, must therefore satisfy certain desiderata which are not incumbent upon a parallel usage in oil and gas bearing formations. Thus, for example, as the coal is burned in final usage, a material previously used in the seam for gas blocking purposes must necessarily not yield noxious combustion products, nor any ecologically unsatisfactory fumes or gases.

Confronted with the described concerns as to the conditions and criteria which must be satisfied or met in order to successfully obstruct gas migration in coal by the use of some type of gel, we have undertaken the testing of silicic acid gels for this purpose, and have realized results establishing the feasibility and advantage of this procedure.

A broad object of this invention is to provide a method for reducing the emission of methane gas from a coal seam at a mining face or rib face within the seam.

A more specific object of the invention is to provide a method of implacing a gel in the cracks and fissures in a coal seam so as to prevent migration of methane gas through such cracks and fissures.

Another object of the invention is to provide a safe and economical method of sealing a coal seam against gas emission over an extended period of time, which method is sufficiently simple that it may be practiced by mining personnel without special education or unusual technical skills.

A further object of the invention is to provide an effective gel barrier in cracks and fractures in a coal seam and, in the course of providing such barrier, to reduce the extent to which coal dust is released from the coal in the course of mining.

Other objects and advantages of the invention will become apparent as the following detailed description of preferred embodiments of the invention is read in conjunction with the accompanying drawings which illustrate the invention.

BROAD DESCRIPTION OF THE DRAWINGS

FIG. 1 is a graph showing the relationship of SiO_2 concentration and pH to gel formation in the case of certain silicic acid compositions.

FIG. 2 is a diagrammatic illustration of a six entry heading in a coal mine in which a field test of the invention was carried out.

FIG. 3 is an enlarged diagrammatic illustration of the placement and directing of injection holes in relation to the rib face, and to the face cleats, in the coal seam constituting the situs of the heading shown in FIG. 2.

FIG. 4 is a composite graph illustrating the effects of silicic acid gel infusion on methane content of air directed across the treated section of the rib face.

FIG. 5 is a composite diagrammatic and graphic illustration of the situs of a second field test of the present invention, and showing graphically the results obtained in such field test.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS OF THE INVENTION

INJECTED GELABLE COMPOSITION

It is believed that the present invention resides, in part, in the recognition of certain preferred and optimum compositions for use in the sealing of the fracture system of the coal, and in part in the recognition of the steps and techniques employed in the desired location in the seam. The compositions will first be dealt with in detail.

A number of properties should desirably characterize the composition which is injected into the formation for creating the gas block in the manner hereinbefore described. Some of these properties are merely to be preferred, and others are of significant criticality to the

successful practice of the invention. The composition, when made up, must be pumpable in order to facilitate its introduction into the coal seam. Preferably, the composition has a viscosity at 70°C which does not exceed 6 centipoises, and most preferably, the viscosity of the composition simulates that of water. In this way, the composition can be pumped easily into the formation, and in commencing to set up to a gel at a later time, will at least have sufficiently low viscosity up until a critical time of gelling that it can move into and fill relatively small cracks and fissures in the formation.

The composition, as injected, should be free of any solid particles of significant size. Although, in some instances, it may be desirable for certain purposes to entrain very small solid particles as a suspension or dispersion in the composition, any solid particles which exceed about 20 microns in size should be avoided. This prevents choking or clogging of certain fractures or pores in the formation so as to require increased pump pressure to force the composition farther into the formation, and past the obstruction thus formed.

A critical property of the composition injected into the formation for the purpose of sealing the seam against gas emission at the face thereof is that the composition have the capability of setting to a gel of sufficient strength that the gel will not yield and thus be bypassed under the gas pressure differential encountered in the formation. With some types of coal seams, the gel which is developed must have a sufficient strength to withstand differential pressures of at least 600 psi. This strength of the gel must, of course, be displayed even in those instances where the gel must bridge across relatively wide fractures or cracks in order to establish an effective barrier in such cracks.

Another critical property of the composition injected into the coal seam for the purpose of preventing migration of gas to the surfaces thereof is that the composition not undergo gelling or set up to a hardened or semi-solid state until it has moved to the desired location within the seam at which the gel barrier is to be formed. While this characteristic of the composition is essential, it is further highly desirable that the composition be susceptible to tailoring of its gelling time in the sense that it can be made up prior to injection with a selected, characteristic gelling time which can be varied as differing requirements are encountered with different types of mining operations in various types of coal.

Related to the gelling time of the composition, and the ability to tailor or control this parameter as may be desired, is the further preferred property of not commencing to undergo any significant increase in viscosity until the composition is very near the location where the gel formation is desired. In other words, the formation of the gel must occur very rapidly once it is started, and it is desirable that the viscosity of the composition, as injected, remain relatively low so that flow of the composition into the farthest reaches of the seam where the gel is to be implaced may occur without impediment or excessively high frictional resistance.

As a final property which should characterize the composition employed in establishing an effective gas block within the coal seam, such material preferably does not yield or give off any noxious fumes, toxic combustion products or atmospheric contaminants at the time that the coal product is burned. Moreover, it is, of course, to be preferred that the composition injected into the seam not be corrosive, and not constitute a hazard or danger to the workmen responsible for its handling and injection.

A composition which meets all of the described criteria and desiderata is a silicic acid colloid which is setable to a silicic acid gel in the coal seam. These compositions are generally derived from the reaction of an acidic material or certain types of salts with a water soluble silicate solution in accordance with reaction mechanisms now well understood in the art. Although silicic acid colloidal dispersions of the type described constitute the preferred gelable material to be injected into the coal seam, other setting or gelable compositions having the described critical properties and preferred characteristics can also be used, and are envisioned as being within the spirit and scope of the invention.

To consider further the preferred silicic acid gels, operations of forming the described gas barrier in coal seams will generally require that the gelling time from initial mixing be at least 15 minutes. In the majority of subterranean mining operations, a range of from about ½ hour to about 12 hours will encompass the gelling times which will be satisfactory. Most preferably, the silicic acid composition will have a gelling time of from about 1/2 hour to about 2 hours in the majority of those instances where the gel is injected through horizontally extending holes into a mining face, or the face of a solid rib, for a distance of from about 60 feet to about 120 feet into the coal seam, as hereinafter described. The shortest gelling period which can be employed, and yet allow complete injection of as much material as desired without the use of unacceptably high pump pressure is the most preferred gelling time in any given instance. This practice avoids "slumping" of the low viscosity, ungelled material once it is implaced at the desired location in the formation. If the gelling time is too short, of course, the result is fracture blockage and impairment of complete injection due to the premature setting up of the gel.

The time which is required for the silicic acid composition to gel or set after it is prepared is dependent upon several variables, including the pH of the compositions when mixed, the effective concentration of silica in the mixed composition, and the character and amount, if any, of any additives employed in the composition. By the proper choice and control of these variables, the setting or gelling time of the composition can be varied from almost immediately after mixing, to an infinite time (no gelation).

Before discussing specifically certain preferred pH and silica concentration values to be used in preparing the silicic acid colloidal dispersions preferably used in the invention, it may be commented that such dispersions cannot be prepared by mixing the reactants used in preparing the dispersions to attain a solution having a pH lower than about 2 or higher than about 12. Moreover, at concentrations of silica of less than about 0.25 weight percent in the composition, a gel either does not form, or forms in an unsatisfactory manner. These limitations therefore establish the outside limits of pH, i.e. from about 2 to about 12, and the lower limit of concentration, i.e. not less than about 0.25 weight percent, which characteristics must characterize the silicic acid compositions of the invention.

Dealing first more specifically with the preferred pH of the silicic acid compositions useful in the invention,

it has been determined that when a pH of from about 6.0 to about 7.5 is utilized, very rapid gelling of the solution will occur, and it is therefore preferred to use a pH either below about 6.0 or about about 7.5. On the other hand, at pH values less than about 2.6, occurs so slowly that for most usages, the time required to establish a sufficiently strong and useful gel for functioning in the present invention is unacceptably long, and moreover, the concentration of SiO_2 in the aqueous composition must be relatively high, i.e., above about 1.0 weight percent in order to yield a satisfactory gel at the most satisfactory pH values. The same is true when the pH of the silicic acid solution, as initially made up, exceeds about 10.7. It will thus be seen that the optimum pH ranges which characterize the useful silicic acid compositions of the invention include two ranges — the first being from about 2.6 to about 6.0, and the second being from about 7.5 to about 10.7. Formulation within these ranges will usually yield gels within the previously identified preferred time periods, provided that the silica concentration in the composition is at least about 1.0 weight percent. The most preferred pH range is from about 7.7 to about 9.9, and the most preferred range of silica concentration in the composition is from about 1 weight percent to about 10 weight percent. It is preferred to use gels as low in silica concentration as can be effectively employed in order that the process of the invention may be practiced more economically

From the curves reproduced in FIG. 1 of the drawings, the inter-relationship of SiO_2 concentration and pH in silicic acid compositions derived from sodium and potassium silicates may be perceived. The curves are substantially symmetrically placed with respect to an abcissa line corresponding to the pH 7 neutral point. As the pH of the gelable composition is moved upwardly or downwardly from this neutral point toward the curve bounding the area in which a gel is formed, the time required for gelling or setting up is increased.

The relationship of the pH and SiO_2 concentration to gel formation was further confirmed by several laboratory tests. In the first of these, a gelable composition was prepared by slowly adding 20 ml. of a sodium silicate solution containing about 9.6 weight percent SiO_2 to 80 ml. of 0.75 N hydrochloric acid. The colloidal dispersion thus prepared, and having a pH of about 1.0, was then divided into five parts, each containing about 1.92 weight percent SiO_2. Concentrated sodium hydroxide was then added slowly, with stirring, to each dispersion to form mixtures having pH values of from 2.6 up to 5.2. The setting time of each of these five colloidal dispersions was then determined. The results are shown in Table I.

TABLE I

No.	pH	Setting Time
1	5.2	less than 10 minutes
2	4.7	about 30 minutes
3	4.2	about 2 hours
4	3.5	about 12 hours
5	2.6	sets into a soft gel after 6 days

As shown in Table I, the optimum pH of the colloidal dispersions made in the manner described is from about 3.5 to about 4.7.

In another series of experiments, a series of dispersions containing SiO_2 in concentrations ranging from 0.4 weight percent to 4.8 weight percent were prepared. In the preparation, the sodium silicate was initially added to the sulfuric acid to bring the pH to about 2. NaOH was then slowly added until the desired pH for the test was attained. The pH of the several solutions thus prepared was adjusted to 4.7 by the addition of sulfuric acid. The setting times for these mixtures are shown in Table II.

TABLE II

Solution and Percent SiO_2	Setting Time at pH = 4.7–4.8
1 (4.8% SiO_2)	Approx. 25 Min.
2 (3.6%)	do. 40 Min.
3 (2.4%)	do. 135 Min.
4 (1.2%)	do. 30 Hrs. (soft gel)
5 (0.8%)	do. 48 Hrs. (soft gel)
6 (0.4%)	More than 7 Days

The results set forth in Table II show that increasing the SiO_2 content of the gelable silicic acid composition results in a decrease in the time required for setting up or gelling. It is further apparent that in the case of the colloidal dispersions having a pH of 4.7, the optimum silicate concentration to be utilized is between about 1.2 weight percent and about 4.8 weight percent SiO_2.

A number of materials can be employed in formulating the gelable silicic acid compositions constituting the preferred compositions used in the method of the invention. A water soluble silicate is mixed with an acid or acid salt to form the orthosilicic acid colloidal suspension. Various organic and inorganic acids can be mixed with the silicate solution, and certain acid salts can also be employed for reacting with the silicate to form the colloidal suspension of orthosilicic acid. Among the acids where can be used in formulating the gelable composition, hydrochloric acid, sulfuric acid, sulfamic acid, propionic acid, nitric acid and acetic acid may be mentioned as exemplary. Acid salts which can be employed are sodium bisulfate, sodium bicarbonate, and ammonium bicarbonate. In general, any reactant which ionizes to yield hydrogen ions which then react with the water soluble silicate to form the silicic acid colloidal suspension can be utilized.

The particular silicate which is employed in preparing the composition can vary widely, and both organic and inorganic water soluble silicates are useful. Because of its relatively low cost, sodium silicate constitutes the preferred silicate used in preparing the gelable compositions used in the invention, and commercially available sodium silicate solution, having an SiO_2 to Na_2O weight ratio of 3.22:1 and containing 38.3 weight percent of sodium silicate in water is a suitable silicate which can be beneficially utilized.

As has been previously mentioned, the chemistry of the orthosilicic acid gels and the colloidal dispersion precursors thereof is generally well understood, and additional materials for preparing such gels, other than those suggested by way of example herein, will be discernible by those skilled in the art. It may be commented, however, that the particular method of formulating the compositions, and the reactants utilized in such formulation, is of some importance with respect to the ease with which the present invention may be

practiced, and the effectiveness of such practice. Thus, it is necessary, of course, to employ pumps and suitable conduit or piping to inject the gelable composition into the coal seam for the purpose of establishing effective gas blocks. It is therefore desirable that the composition have minimum corrosivity with respect to the pumps, piping and other metallic elements of the system used for injection. It is thus of advantage, all other considerations aside, such as the great economic advantage of sulfuric acid, to utilize a relatively weak acid or acid salt to react with the water soluble silicate, and to prepare gelable compositions in the basic pH range hereinbefore described. It is also desirable to use a salt which produces a relatively low number of hydrogen ions per unit volume upon ionization. In this way, better control of pH and ultimate setting time can be attained as a result of the relatively small effect upon pH and setting time which may occur if errors in volumetric measurements, leakage or the like should occur in mixing.

A particularly desirable salt utilized in making up the compositions of the invention is sodium bicarbonate, since this material is relatively inexpensive, can be added in fairly substantial quantities in an aqueous solution without producing a wide variation in the pH of the product composition, is not corrosive and can be easily handled and blended by mine personnel with little technical training. In the use of this material, and the preferred sodium silicate reactant, a composition is prepared having an adjusted pH which is preferably from about 7.7 to about 9.9, and which has a SiO_2 composition equivalent of from about 1 weight percent to about 10 weight percent. Such compositions will generally set up to a relatively strong gel in from about 30 minutes to about 2 hours, and thus are satisfactory in this respect for most plugging or sealing operations.

In formulating the gelable compositions used in the invention, it is preferred to add the silicate to a strong acid, when such is used, and to carry out the addition rapidly and with vigorous agitation so as to pass quickly through the neutral pH zone. This mixing procedure is highly desirable because of the very rapid gelling which occurs in that range, and the possibility of lump formation occurring during transit of the neutral range. Further, it is preferred that in any instance where the mixing causes the overall pH of the reaction mixture to traverse the neutral pH range, that such mixing be carried out relatively rapidly and with vigorous agitation, so that localized formation of lumps of gel is avoided.

The strength of the gels formed from the preferred compositions of the invention, in the sense of the ability of the gel to block the flow of gas through cracks and fissures, is generally more than adequate to establish an effective block against the methane pressures existing in coal seams. These pressures vary widely, and it has been reported that the pressure of natural gas in the Pittsburg coal bed can reach 180 psi, and in other reported seams can reach as much as 600 psi. Gels prepared from the compositions herein described have been tested for strength in various types of sandstone cores, as well as in coal fractures, and have demonstrated strengths sufficient to hold differential pressures in excess of 1,500 psi. The strength of the particular gel block, in terms of its ability to hold the gas in place rather than permitting it to by-pass the plug, will, of course, depend upon the size of the fracture or crack which must be sealed, and the distance which the gel plug must bridge.

The viscosity of the gelable silicic acid compositions prepared in accordance with the foregoing discussion is generally quite favorable to pumping of the colloidal dispersions into the seam. Generally, an initial viscosity well below 6 centipoises is characteristic of the compositions upon initial formulation, and the viscosity remains below about 5 centipoises over at least ¾ of the time period required for the final gel to set up in the seam after the time of mixing. Thus, pumping of the compositions is easily accomplished until the amount of the composition to be injected has entered the seam.

METHOD OF INJECTION OF THE GELABLE COMPOSITION

In the subterranean development mining of coal seams, a widely used practice is the extension of multiple entry headings in a generally horizontal direction into the seam. The seams may vary in their vertical thickness from about 1 foot in height up to about 9 feet in height. As the headings are extended into the seam, there are formed at opposite sides of the heading, vertical faces of solid coal ribs which extend along the length of the heading. At the forward end of the heading in the direction in which it is progressing, a working or mining face is generally formed as the coal is removed from this location. Dangerous quantities of methane are often emitted from the rib faces and from the mining or working face.

Depending upon the cleavage and bedding plane orientation in the particular seam under production, a heading may be extended at any one of several angles with respect to the face cleats or planes of cleavage within the coal. Many seams of bituminous coal are characterized by well defined face and butt planes of cleavage, and clearly perceptible, well defined face cleats and butt cleats. It is through the cracks or fissures which exist at these locations that methane gas, desorbed from the micropore spaces within the solid coal, bleeds to the faces bounding the heading, and is there emitted to constitute a danger to personnel located in the mine.

In the practice of the process of the present invention, substantially horizontal infusion holes are drilled into the coal seam from a rib face or mining face of the heading, and are preferably drilled in a direction to intersect face cleats of the coal at a right angle. This orientation is utilized because the face cleats are generally regular in their orientation with respect to each other, and extend in substantially parallel, vertical planes in the seam. By extending the infusion holes which are drilled for the purpose of injecting the gelable composition into the seam in a direction such that the holes intersect the face cleavage planes at a right angle, more of these planes are cut across by the hole per linear unit of hole distance, and the composition can thus be placed in more of the cracks and fissures occurring at the face cleats by injection through a relatively shorter hole. For example, where the direction in which a heading is being extended into the seam cuts across the face cleavage planes at a 90° angle, then it is preferred to extend the infusion holes into the rib face along the sides of the heading at an angle which is as shallow with respect to the face of the rib as possible. On the other hand, where sealing of the mining face at the forward

end of a heading advanced perpendicular to the face cleavage is to be carried out, the injection holes are preferably extended normal to the mining face, and therefore normal to the face cleavage planes of the seam which extend parallel to the mining face.

The number of holes which are drilled into a rib face or mining face for the purpose of blocking the flow of methane gas thereto, and the depth to which such holes are drilled, will, of course, depend upon the fracture porosity of the particular seam being treated, the cleavage development of the seam, the gas pressure in the particular seam and the properties of the composition being injected. The general desiderata which must be met is that a sufficient areal surface along a rib face or mining face be protected by the blocking and diverting effect of the gel that mining can be continued without concern for excessive emission of methane gas at the working location. This will, in many instances, also depend, of course, on the type and efficiency of the air circulation and ventilation system which is in use.

In those instances where infusion holes are being drilled at an angle to a rib face along a heading, a preferred technique for placement of the holes is to extend one of the holes into the seam in the general direction in which the heading is being advanced, and at an angle such that the face planes of cleavage are penetrated by the hole in as nearly a perpendicular direction as possible. There is then cut, in advance of the hole so formed, a second hole sufficiently advanced along the face of the rib that the second hole, when extended at approximately the same angle, but in a reverse general direction into the seam from the first hole, will cross or intersect the vertical plane containing the first hole at some location near the maximum depth into the seam to which the first hole has been projected. In other words, a series of shallow V's are formed along the rib face to be sealed by adjacent pairs of holes formed therein. This has been found to give good coverage and protection to the rib face, provided the holes are drilled to sufficient depth. In general, the holes employed for injection can be extended into the seam in the course of sealing of a rib face for distances of from about 50 feet up to about 200 feet. Where the fracture porosity is good and butt cleating is well pronounced affording communication between adjacent planes of face cleavage, the depth to which the hole is extended can be less than in those cases where such high fracture porosity does not exist. In the latter case, the hole which is drilled must be primarily depended upon for conveyance of the gelable composition to very substantial depths within the seam, for the butt cleat communication system does not adequately serve this purpose.

In the case of sealing of a mining face, the injection holes will generally be drilled to a substantially greater distance into the seam than will be necessary or desirable in the case of the sealing of rib faces. This is true because, as the mining is carried on to advance the heading, a portion of the effective block formed by the injected gelable composition is being continually removed, and the effective pressure differential of entrapped gas acting across the remaining gel ahead of the advancing mining face is increased. Thus, by drilling the injection holes to a greater distance into the seam ahead of the mining face, the frequency with which mining must be terminated to re-treat the formation is lessened.

The silicate and acid components which are blended to formulate the composition to be injected into the infusion holes are preferably maintained separate and not blended until very shortly before injection is to be commenced. It is, moreover, not desirable to blend and formulate large quantities of the gelable composition in one container, since the period of time required to complete the injection of such large quantities may be sufficiently extended that some incipient setting up or significant increase in the viscosity of the gelable composition may occur.

As the composition is injected into the formation, it is preferable to maintain relatively high pressure on the material being injected until immediately prior to the time when the injection is completed. This will assure that the gelable composition which has been placed in the cracks and fissures where gel formation is desired will not slump or gravitate in retrograde flow within the crack or fissure as pressure from the injection face inwardly in the seam is relieved or lowered. Generally, pump pressures utilized with the preferred silicic acid gels of the composition should be at least 50 psi. Pressures in excess of 150 psi are to be avoided, however, so as to avoid any possibility of enlarging or widening the fractures or pores in the seam, and thus aggravating the gas seepage and emission problem.

The quantity of the composition injected will depend upon the magnitude of the fracture porosity, and, of course, the depth to which injection is to be carried out. It is generally desired to inject a sufficient quantity of the composition to completely fill the fracture porosity over a vertical distance equivalent to the distance between the floor and roof of the mine, and a horizontal distance which is sufficient to fill all of the cracks and pores of the seam existing over a distance along the rib face corresponding to the horizontally projected distance of the infusion hole in the plane of the rib face. With the holes overlapping and criss-crossing in the manner hereinbefore described, assurance can usually be had of complete horizontal coverage along the rib or mining face.

A more comprehensive understanding of the method of the present invention, and of certain preferred embodiments of its practice, will be gained from the following examples which describe certain field tests of the invention which have been carried out.

EXAMPLE 1

Gas emission has been encountered in the Blacksville No. 1 mine of Consolidation Coal Company. Emission of methane at the rib faces along the 4-North heading in that mine has been significant in the past, and has required the shutdown of mining operations at the mining face within the heading. The 4-North heading is a six entry heading, and its general appearance is shown in FIG. 2 of the drawings. The direction of air flow in the heading is shown by arrows, and it will be noted that air is coursed in through the No. 6 entry and is then divided between two entries (No. 5 and No. 6) along a major portion of the east rib of the heading. The heading extends in a generally northerly direction and crosses the face cleavage of the coal seam at an angle of about 73°. The site in the 4-North heading selected for the tests of the effectiveness of the silicic acid gel in sealing a local section of rib 20 had about 800 feet of clear rib face exposed between two falls and experiencing, at the time of the test, a significant increase in

methane emission from one end of the exposed section of rib face to the other.

Prior to setting up and commencing a field test of the present invention in the 4-North heading of the Blacksville mine, plastic curtains were rigged across the No. 5 entry to divert all of the air flow from that entry into the No. 6 entry adjacent the rib face into which the injection holes used in the field test were to be formed.

The equipment utilized in the field tests in the Blacksville mine was mounted on two flat cars movable along the trolley track in the heading. One of the flat cars carried the mixing and pumping equipment, and one carried drums of sodium silicate and bags of sulfamic acid to be used in formulating the gelable composition. Two cut-down 55 gallon drums were used to make up, in each drum, 40 gallon batches of the gelable silicic acid composition. Suitable plumbing was arranged in conjunction with a triplex injection pump so that the pump could take suction from either one of the mixing drums while a new batch of the composition was being mixed in the other. The measured maximum output of the pump was 14.8 gallons per minute. Water from a belt line in the mine was piped to the mixing drums, and valving was provided to allow water to be directed into either mixing drum, or pumped directly into the infusion holes.

The general procedure employed was to drill a series of infusion holes to a distance of 70 feet into the rib face, criss-crossing the holes as shown in FIG. 2 of the drawing, where the infusion holes are designated by reference numeral **22**. As shown in FIG. 3 of the drawings, the infusion holes **22** were drilled at an angle of about 27° to the rib face. Those holes projected in the general direction of extension of the heading, and angled in this manner relative to the plane of the rib face, intersected the vertical planes of face cleavage at an angle of about 90°. In this way, a greater number of the vertical face cleavage planes are intersected by a hole drilled to a given distance within the seam. The holes drilled in the generally reverse direction from the direction of extension of the heading are drilled to a depth, and at an angle, such that the vertical plane of the adjacent forwardly extending hole is intersected, and the depicted criss-cross pattern results.

A total of 14 of the 2 inch holes **22** were drilled in seven criss-crossed pairs along the rib face as shown in FIG. 2 of the drawing.

The infusion pipes used to direct the composition into the infusion holes after they are drilled extend for a distance of about 20 feet into the hole, and a packer, included on this pipe, is set to plug the hole about 12 or 13 feet into the hole from the opening thereof at the rib face. As soon as the drilling of any hole was completed, and before drilling the next hole, the injection equipment was set up and first water and then the gelable composition were injected into the hole.

Preparatory to injecting the composition into the seam, the first 40 gallon batch of the composition was made up by adding 9.5 pounds of dry sulfamic acid to 33.4 gallons of water in one of the 50 gallon drums. After complete solution of the acid in the water, 6.6 gallons of a commercially available sodium silicate having an SiO_2 to Na_2O weight ratio of 3.22:1 were added to the acid solution with concurrent mixing, and mixing was continued for one minute. The mixer was then turned off and injection was commenced. A control sample of the silicic acid composition was placed in a beaker and set aside for observation. This method of preparing each 40 gallon batch of the composition was repeated as needed for each injection.

As indicated above, water was injected into each hole at a pressure of 100 psi in an average rate of about 0.3 gallons per minute per foot of hole in the several holes prior to the time that the silicic acid composition was injected. The purpose of the water injection was to gain additional information on coal seam permeability and porosity by observation of seepage of water at the rib face, and to estimate the probable gel infusion rate for the injection of the gelable composition to follow.

During and after completion of the injection of the silicic acid composition, the face of the rib, and the roof and floor of the heading were watched for the appearance of water or gel. The blocking plastic curtains, which were hung in the No. 5 entry to divert air down the outside (No. 6) entry during the course of the test, permitted measurements of the methane content of air moving past the infusion holes progressively drilled in the rib face, and further periodic measurements of the methane content of the air passing the downstream end of the 800 foot test span of the rib face. The change in methane concentration of the air over these intervals was considered to be a measure of the effectiveness of the silicic acid composition in establishing a seal against methane flow to the rib face. The methane concentrations were also recorded at a number of points along the test section of the rib each day that the infusion and sealing work was being done.

The observed results of the water injection tests which preceded injection of the gelable composition are summarized in Table III. This table also sets forth the observations of water appearing at the rib face and roof following the injection of the silicic acid composition. The appearance of water from the lower part of the rib to as much as one-half way across the span of head coal in the roof over the No. 6 entry indicated good vertical coverage of the rib face by the injected materials, and indicated that the water and gelable composition appeared to move very freely along the face cleavage of the seam.

TABLE III

Hole No.	Total Depth, Feet	Water Inj. Rate, GPM	Water* Inj. Press, psig	Rate Per Ft. of Open Hole, GPM	Remarks Concerning the Appearance of Water in the Entry During Injection
5	51	13.4	130	0.34	Water both from rib & over ½ way across roof. Extends for 60 ft. inby No. 5 hole at rib.
6	70	—	—	—	No noticeable water.
7	70	22.2	100	0.38	No noticeable water.
8	60	8.8	100	0.18	Some water out of roof & rib for about 65 ft from hole in rib.
9	70	18.6	100	0.32	What appeared to be gel out of rib over distance of about 30 ft from hole. No evidence of water for rest of hole length.

TABLE III—Continued

Hole No.	Total Depth, Feet	Water Inj. Rate, GPM	Water* Inj. Press, psig	Rate Per Ft. of Open Hole, GPM	Remarks Concerning the Appearance of Water in the Entry During Injection
10	70	11.3	100	0.20	Water out of rib & roof for 112 ft from hole. Gel (?) came out for about 65 ft.
11	50	11.3	100	0.30	Water out of rib for 35 ft from hole & gel (?) for 22 ft.
12	70	13.0	100	0.22	
13	70	13.4	100	0.23	Water out of rib near floor for 30 ft from hole.
14	70	25.4	100	0.44	Water from rib at only 1 spot which was about 30 ft from its hole.
17	70	20.0	100	0.35	
16	70	25.4	100	0.36	Water out of rib for 50 ft from hole.
17	52	15.5	100	0.39	Water from rib for 25 ft from hole.
18	70	8.0	100	0.14	

*At pump

The volumes of the composition infused via each of the holes are shown in Table III. The weighted average in the case of each of the holes was an injection of 3 gallons of the composition per foot of the open hole between the packer and the end of the hole. Assuming a crack porosity in the seam of 1 percent, this amount of gel injected would have been adequate to fill out a cylinder of 7 foot diameter around the infusion hole.

The effectiveness of the seal established by the gel upon injection into the seam is shown in FIG. **4** of the drawings. This is a graph illustrating the increase in methane concentration in the air along the test section of the rib. The methane was measured as weight percent in the air, but all the curves in the graph have been normalized to a base value of 1.0 to eliminate anomalous ups and downs in the curves caused by changes in the volume of ventilating air entering and passing by the test section of the rib.

A first curve is plotted for data taken on Dec. 8, 1971 prior to the commencement of the infusion tests. A general increase in the methane content of the air along the entry bounded by the rib face was noted, and the overall increase is 0.26. Data for a second curve were taken on the 21st and 22nd of December, 1971 after about 300 feet of the 800 foot test section of the rib had been infused with the silicic acid composition in the manner described. The curve shows some flattening in the infused area, indicating a decrease in methane content at this location, and the overall increase over the test section is considerably less. The third curve is based upon measurements taken on Dec. 29 and 30, which measurements were averaged, and which were made when the infusion holes had been placed in the rib for a distance of about 500 feet therealong. These measurements showed that the methane was definitely sealed off at the upstream or origin end of the test section. The fourth curve depicted in FIG. **4** is based upon methane measurements taken on Jan. 11, 1972 and shows that by this time, the methane has broken back through the gel at the origin of the rib test section. The advanced or downstream end of the section, however, appears to be sealed substantially better than prior to the commencement of the test, as indicated by the December 8th curve.

In summary, it appears that in the field test carried out in the Blacksville mine in the manner described, the seal established was effective for a period of about two weeks. This was a shorter time than was deemed optimum. The methane gas, when it broke back through the seal as indicated by the January 11, 1972 measurements, appeared first from the head coal in the roof of the heading, and from the upper part of the rib. This suggests that a less than optimum volume of the gelable composition had been injected into the seam for purposes of obtaining complete vertical coverage. The volume to be injected in any seam of unknown fracture porosity is necessarily empirically determined, and in this test, a larger volume of injected composition would probably have produced a longer lasting seal.

EXAMPLE 2

A second field test of the invention was carried out in the 12-North heading of the Osage No. 3 mine of Consolidation Coal Company. The test covered 1,000 feet of solid rib near the inby of the heading. A multiple entry heading was again involved, and the arrangement is that depicted at the bottom of the composite illustration in FIG. **5** of the drawing. The symbols referred to in the legend accompanying FIG. **1** are also applicable to the illustration of the 12-North Osage mine heading shown in FIG. **5.** The 12-North heading at the Osage mine traverses the face cleavage of the seam at about the same angle as the 4-North heading in the Blacksville mine as described in Example 1.

The infusion holes were again extended at an angle of about 27° to the rib face, and paired in opposite directions to form the criss-cross pattern depicted in FIG. **4,** where the holes are designated by reference numeral **30.** The numbered black dots appearing in this depiction of the 12-North heading show the points at which methane monitoring instruments were set up along the rib face constituting the test section.

Prior to the commencement of the test by injection of the gelable composition, readings were taken at 12 test stations on 9 separate days, and these readings were then averaged to obtain a curve illustrative of the methane content of the air along the rib face prior to commencement of testing. This curve has been reproduced in the graph appearing immediately above the schematic illustration of the 12-North heading of the Osage mine in FIG. **5.** The infusion holes used were drilled along the first 800 feet of the rib just inby of the main heading from which the 12-North heading extends, and the test conditions employed in the Blacksville mine field tests, and cited in Example 1, were, in general, repeated. In the Osage mine test, however, the gelable composition used was prepared by mixing sodium silicate with sodium bicarbonate to attain a composition having a pH of about 9.9 and a silicon dioxide concentration of about 2.3 weight percent. In the case of the Osage mine field test, all of the desired predetermined volumes of the silicic acid composition were successfully injected into each of the approximately 70 foot long infusion holes.

Upon completion of the drilling of the holes in the rib face, and the injection of the gelable silicic acid composition into the rib, methane readings were periodically

taken at the twelve testing stations along the rib for a period of three weeks following the tests, and these readings were averaged for comparison with the reading averages taken prior to the commencement of the testing. A plot of the average readings thus obtained along the 800 foot rib face is set forth in the graph appearing in FIG. 5. The averaging procedure utilized eliminated any effect of methane variations in the air entering and flowing by the test section. In the arrangement shown in FIG. 5, the data points on the two curves have been established in vertical correlation with the corresponding test station where the averaged measurements were made, as shown along the rib face of the heading as it appears immediately below the graph.

Reference to FIG. 5 of the drawing reveals that the overall reduction of the methane content of the air along the infused rib section is better than 60 percent. Moreover, plots of the data for individual monitoring stations do not show increases in methane during the three week period following infusion, indicating that the seal remains substantially equally effective over that period following completion of the test. It is believed that the observed lack of total shut-off of methane emission reflects incomplete vertical coverage of the seam by the injected composition. Again, the quantity of the composition which must be injected to achieve complete vertical coverage and optimum sealing must usually be empirically determined due to variations in fracture porosity, and the presence of laminae of clay and other material frequently present in the seam. It will be recognized, of course, that complete sealing is frequently not needed to maintain the methane concentration in the air adjacent the seam well below tolerable limits, and economics may frequently dictate the use of a lesser quantity of the composition than would be needed to obtain the most effective and complete seal.

From the foregoing description of the method of the invention, and the several embodiments representative of its practice which are described and which are illustrated generally by the field test description and data presented, it will be seen that the present invention provides an effective procedure for significantly reducing the emission of methane gas from exposed rib and mining faces developed in subterranean development coal mining operations. The particular compositions which have been described as illustrative of compositions which can be used in the practice of the invention, and the infusion procedures referred to in the description and in the specific examples, can be varied considerably without departure from, or relinquishment of, the basic principles of the invention. Changes and innovations of this type are therefore deemed to be encompassed within the spirit and scope of the invention, except as the same may be necessarily limited by the appended claims or reasonable equivalents thereof.

What is claimed is:

1. A process for reducing gas emission at an exposed face in a coal seam during subterranean mining of the seam by blocking and diverting the gas to a non-worked location in the mine comprising injecting into the seam at multiple locations to enter cracks and fissures at the bedding planes in the coal seam at locations spaced inwardly in the seam from the exposed face, a pumpable silicic acid colloidal dispersion capable of setting up to a semi-solid gel in said cracks at the bedding planes and thereby sealing said cracks and fissures against gas movement, said colloidal dispersion being characterized in having a viscosity, when injected, of less than 6 centipoises, a pH of from about 2.0 to about 12.0 and an SiO_2 concentration of at least 0.25 weight percent.

2. The process defined in claim **1** wherein said composition is injected by:

drilling substantially horizontal infusion holes into the coal seam from said exposed face in a direction to intersect face cleavage planes in the coal at substantially a right angle; and

injecting said gelable composition into said infusion holes.

3. The process defined in claim **2** wherein additional holes are drilled into the coal seam from said exposed face in a direction such that each of said additional holes intersects the vertical plane in which one of said first mentioned holes lies to form a plurality of criss-crossed hole pairs in said seam arrayed in plan view as a series of shallow V's.

4. The process defined in claim **1** wherein said gelable composition is injected at a pressure of less than 150 psi.

5. The process defined in claim **1** wherein an injection pressure equal to at least the initial injection pressure is maintained on said gelable composition until the time when said gel sets up to a semi-solid gel.

6. The process defined in claim **1** wherein said silicic acid colloidal composition has an SiO_2 concentration of at least 1 weight percent.

7. The process defined in claim **1** wherein said silicic acid colloidal composition has a gelling period, as measured from the time of initially formulating the composition to the time it sets to a semi-solid gel, of from about ½ hour to about 12 hours.

8. The process defined in claim **7** wherein said gelling period is from about ½ hour to about 2 hours.

9. The process defined in claim **1** wherein said silicic acid colloidal dispersion is prepared by mixing a water soluble silicate with a hydrogen ion yielding material selected from the group consisting of hydrochloric acid, sulfuric acid and sodium bicarbonate.

10. The method defined in claim **9** wherein said hydrogen ion yielding material is sulfuric acid.

11. The method defined in claim **10** wherein said silicic acid colloidal dispersion is prepared by adding the water soluble silicate to the sulfuric acid rapidly and with vigorous agitation to avoid formation of solid particles of gel during mixing.

12. The method defined in claim **9** wherein said hydrogen acid yielding material is sodium bicarbonate.

13. The process defined in claim **1** wherein said silicic acid colloidal dispersion has a pH of from about 7.5 to about 10.7.

14. The process defined in claim **1** wherein said silicic acid colloidal dispersion has an SiO_2 concentration of from about 1 weight percent to about 10 weight percent.

15. The process defined in claim **14** wherein said silicic acid colloidal dispersion has a gelling period, as measured from the time of initially formulating the composition to the time it sets to a semi-solid gel, of from about ½ hour to about 2 hours.

16. The method defined in claim **15** wherein the pH of said silicic acid colloidal dispersion is from about 7.7 to about 9.9.

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