

# Black powder control starts locally, works back to source

## Part 2: Research finds procedures, equipment to help operators search out and manage iron sulfide

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**B**lack powder (iron sulfide) in pipe lines is a problem that can be profitably managed by diligent operators who determine that they have a problem and learn all they can about iron sulfide formation and handling from the store of knowledge within their own industry and in other industries.

Part 1 outlined the problem facing the industry and presented a preventive list for use as a starting point by pipe line managers who elect to protect their assets. Part 2 describes preventive measures that provide local relief, such as filtering, and procedures that could be adapted to drive a more aggressive program to prevent, or even eliminate, the problem reaching pipe lines.

**Filtering.** The most common means of dealing with black powder is filtering it just before it enters a compressor station or processing plant.

Various filter technologies and names are usually specified and supplied as a part of the compressor design package when the unit is installed. Sometimes purchasers specify filter performance in their bid request. Sometimes the design requirements given are accurate and sometimes they are hardly known. At other times, the conditions change after the initial installation. The net result is that, regardless of what is requested or promised, it usually is impossible to get field measurements to accurately determine filter and separator performance.

The only identified measure of filter performance is an increase in pressure drop measured across a particle-removal device, indicating that it is collecting material and starting to clog up. It is difficult to identify the quantity and

description of what gets through the filter. Ideally, filters are cleaned out or backflushed at optimum points, indicated by AP, to minimize flow resistance, penetration or overflow.

A brief industry review found a number of filter designs for removing solids or liquids from a gas pipe line. Many are designed to trap particular materials and, consequently, are not good for every type of material or service.

The following are some general comments which should be considered in the design or selection of a filter for iron sulfide. Along with the filtration of iron sulfide, filters nearly always need to be able to handle liquids. Note that not all filtration systems are designed with these two components in mind. General filter comments:

- To trap iron sulfide, a filter must be

designed to remove submicron-size particles.

- To trap any significant volume of liquids, the filter must have a volume for catching liquid slugs, as well as a means of dropping out mist.

- A backflushing method for cleaning filters will make the filter last longer between replacements. Although a manual system can have some value, an automatic sensing and actuating system is the best way to prevent the powder from heavily caking so that it cannot be back flushed. However, an automatic system is another maintenance item.

- A filter should have a means of sensing pressure drop or another indication of filter effectiveness and fullness.

- A self-cleaning system needs to be



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fitted with a means for removal of the black powder and liquids. In view of the pyrophoric nature of the powder (when it is dry and exposed to air, it can auto-ignite), the depositories for the powder and the filters should either be fireproof, or the material should be treated to remove its auto-ignition characteristic.

- Simplicity of filter replacement must be designed into the system. Consideration should be given to easy access into the vessel without compromising the pressure boundary during normal operation, minimum time required for removal and replacement of filters, and crew safety during work while the vessel is open.

The latter point could be implemented by using a design that includes a removable filter holder. A second filter holder could be fitted with new filters outside the unit and, once the filter vessel is opened, the whole holder could be changed out quickly with a minimum of tool work.

From an overview perspective of the black powder problem, filtering is a near-term solution. However, under the scrutiny of a root-cause analysis a permanent solution, filtering is a superficial or local spot solution. Filtering would be less demanding and less expensive if the source of the gas were properly treated to eliminate iron sulfide formation.

**Gas components.** In many instances, the trace components in natural gas that have negligible influence on heating value cause the formation of iron sulfide and resulting equipment problems.

Of the generally desirable hydrocarbon components, isopentane and heavier components can drop out as free liquids at some point in the range of normal operating conditions. Liquids of any source are not desirable because they cause equipment problems.

Other naturally occurring or frequently found components are hydrogen sulfide, sulfur, carbon dioxide, water and nitrogen. All occupy space and absorb energy when moved, thus reducing pipe line efficiency, and they either do not support combustion, or they absorb energy when the natural gas is burned. All except nitrogen can form acidic compounds contributing to corrosion.

Some pipe lines reportedly allow as high as 1 grain of hydrogen sulfide per

100 cf.

Carbon dioxide is a strong acid former and can cause significant pipe line corrosion where water dropout occurs. Allowable levels for this component may be as high as 3% by volume.

Water's effects were pointed out in the discussion of MIC. Water also causes corrosion and equipment damage, and, in fact, corrosion cannot occur in the absence of liquid water.

Oxygen is not naturally occurring in natural gas, but can intrude through leaks in low pressure systems. It can cause significant corrosion in very small concentrations and combustion in larger portions. Some companies reportedly allow as much as 1% oxygen. Even when oxygen is not in the free, gaseous state, it can contribute to reactions if it is present in sulfates, nitrates, water, or other oxygen-containing molecules.

The interaction of oxygen, carbon dioxide, hydrogen sulfide, and water in combinations in natural gas pipe lines is discussed in detail by Lyle. This PRCI research recommends that transmission pipe line companies consider limiting maximum oxygen concentrations to 10 ppmv.

With regard to carbon dioxide content in natural gas pipe lines, oil and gas industry experience indicates that significant corrosion of steels can occur at carbon dioxide partial pressures as low as 7 psi, or about 1.4% by volume. No minimum below which pitting will not occur has been established. Normal carbon dioxide gas quality levels are about 2 to 3%. The research indicates that in the presence of oxygen, water, and carbon dioxide, high concentrations of hydrogen sulfide in excess of all gas quality specifications (maximum of 1 grain per 100 scf) can retard corrosion, but this is not a practical consideration. In these tests, carbon dioxide seems to be a corrosion accelerator. Other combinations of these components were investigated.

**Testing.** There are several tests that can be conducted on a gas pipe line to provide useful information on black powder formation. Among these are gas composition, water content testing, powder composition, and location or origin testing.

Sulfate-reducing bacteria (SRB) produce short-chain fatty acids, as well as carbon dioxide, hydrogen, methane, and hydrogen sulfide. Since the gases in this list are also components in natural gas,

testing for their presence can only provide supportive evidence of microbial activity. Testing for short chain fatty acids is indicative of current biological activity. These acids can be detected using a variety of methods which include ion and gas chromatography and high-pressure liquid chromatography, but these are expensive tests which must be performed by an experienced person, and not likely in the field.

Gas composition testing involves collecting a gas sample from a specific location in the pipe and testing for its constituent gases. Normally, liquids and, particularly, solids are not collected. Many gas composition tests look only for hydrocarbons and a few other common constituents to verify compliance with heating value specifications.

However, it is well-known in technical circles that sulfur, hydrogen sulfide, oxygen and water vapor, some in parts per million, can be important in the formation of iron sulfide and other corrosion products. As discussed earlier, the presence of these components in small quantities can provide:

- Constituents for chemical formation of iron sulfide
- Environment for growth of SRB and acid-producing bacteria (APB) whose metabolic processes result in the production of iron sulfide
- Direct corrosion of steel by oxygen, carbon dioxide, or combinations of the two.

Therefore, it is important to test gas composition samples for these lesser components, to monitor the likelihood of iron sulfide formation. To the extent possible their presence, particularly water, hydrogen sulfide, carbon dioxide, and oxygen, should be controlled within the limits discussed and within the rationale for limits that are being sought by continuing research.

#### **Gas composition enforcement.**

Where content limits are specified in tariff restrictions, they should be enforced at custody transfer points. This effort to identify and remove the small quantities of these iron sulfide producing chemicals may be the most effective approach to preventing the formation of iron sulfide and prevent the damage it causes. Again, the econ-

omics of this effort should be weighed against the total impact of the presence of iron sulfide and MIC in an entire pipe line system.

Even within the limits of these reasonable restrictions on trace components, consider the following mass balance: hydrogen sulfide at 1 ppm (0.25 grains per 100 cf is 4 ppm) in a continuous gas stream of 10 MMcfd, if all converted to iron sulfide, will produce more than 800 pounds of FeS in a year. Thus, even gas meeting many H<sub>2</sub>S specifications can produce large amounts of iron sulfide.

**Water testing.** Water is an important factor in the support of environmental conditions necessary for microbes to thrive and multiply. It is also a factor in direct oxidation of the walls of carbon steel pipe and MIC cannot occur in the absence of liquid water. As discussed earlier, most pipe lines have some water content, and changing atmospheric temperature or other pipe line environmental temperatures can cause water vapor to quickly condense in the pipe and gravitate to the lowest point. For this reason, Canadian gas transmission companies reportedly have lower allowances for water content than companies in other countries, particularly in the more temperate zones. This investigation did not have the opportunity to determine whether they also have lower incidence of iron sulfide. If water is not drained off frequently, it can cause the microbial spores which may be dormant in the pipe, to bloom grossly and rapidly and cause MIC.

SRB and APB prefer the interface between a water puddle and a drier area. They create what is called a biofilm. Thus, the most likely locations to find MIC is at low places in pipe where water is likely to exist and to collect. Eliminating or frequently draining these sites is desirable to avoid pipe damage and iron sulfide creation.

Since the biofilm is the best site for microbial growth, it is also the best medium for identifying their existence. Ideally, the water should be sampled without exposing it to air and it should be tested before the microbe count has a chance to change significantly. This implies using a small sample tube to expose the water to as little air as possible coming out of the valve. The tube should fill the collection vessel

from the bottom to overflowing so that there is no air space. The vessel should be capped or valved off immediately. This nearly air-free sample should be tested within the hour for SRB or APB. More practically, the test is usually performed within 24 hours. Five to 10 ml of water is sufficient for testing for both aerobic and anaerobic bacteria. A good sample vessel might be designed somewhat like a gas sample bottle with inlet and outlet valves on opposite ends. It should be sanitized and then purged with nitrogen or natural gas.

It is often difficult or impossible to sample and test in the field with a high degree of detail. In this case, as much care as possible should be taken to minimize oxidation or contamination of the sample and to reduce the time before it is tested. Some useful results may still be achieved from samples collected and tested somewhat outside of these parameters.

The water sample should be tested for microbial content and the type determined by culture. Such tests should be done by a qualified laboratory. However, on-site test kits are commercially available. They permit testing to be performed by trained gas company personnel. A reduced pH of the water can indicate APB activity.

If there is a low place in the pipe that collects water and there is a way to get the water out, it should be removed frequently to reduce the chances for corrosion, microbial blooming and biofilm growth. Testing for microbial content should be a secondary effort. Therefore, all water should be removed whenever samples are taken for testing.

**Powder composition testing.** When black powder is found in pipe, instrumentation or compression equipment, sometimes it is helpful to verify its composition by chemical testing to identify or verify its likely origin. The origin could be chemical combination or microbial corrosion. There are dark materials that can occur from other materials or chemical reactions that may not be iron sulfide.

Certain forms of iron sulfide are pointers to MIC presence in a pipe line. Iron sulfide forms, mackinawite, smythite and greigite, are considered to be indicators of microbial activity in pipe. The most positive indication of MIC is the presence of pits in the pipe wall. Another is testing to confirm the presence of SRB or APB.

The activity of APB can be identified from the presence of short chain fatty acids such as acetic, formic, lactic, butyric and

valeric acids. Due to the symbiotic coexistence of APB and SRB, if these short chain fatty acids are found, there is good reason to expect the presence of SRB.

**Origin testing.** Testing methods may be used creatively to aid in vectoring the source of MIC or sulfide entry into the pipe line.

In cases where hydrogen sulfide or other sulfides enter a pipe line, direct chemical conversion to iron sulfide will likely take place. This problem occurs most commonly in gathering lines where gas comes directly from wells and where gas flows from several wells are added together before becoming or being added to a transmission line. Once sulfide enters the pipe line at any point, conversion to iron sulfide is prompt. The material can progress down the pipe line from that point.

In the case of MIC-generated iron sulfide, a similar phenomenon occurs. When the components that promote microbial growth get together, a colony and a biofilm are created. Black powder can migrate from such points to locations downstream in the pipe line.

When black powder is found at any point in the pipe line, such as a filter or compressor, it is helpful to be able to identify the source of the material in order to minimize or eliminate it. Performing the above gas, water or powder testing at accessible ports in the pipe may provide clues to the source by identifying where the powder is or is not and whether it has MIC signatures or not. If the ability exists to inspect inside the pipe wall, the existence of wall pitting is a positive indication of a MIC source. If gas testing shows sulfides, it is likely that black powder exists or will be formed soon downstream from that point in the pipe. Water testing should indicate either MIC presence or the likelihood of near-future formation at that point.

Finding iron sulfide powder at a location in a pipe, without other indicators, is not definitive as to whether the powder is forming locally or has moved from upstream. Identifying an upstream location without black powder would help to vector to the source. It does not aid the vectoring process if the powder is being formed at a number of sites near the gathering

portion of the pipe line. Appropriate sites for performing gas collection, water draining or powder inspection and removal are not always conducive to effective powder source vectoring.

If an operator should be fortunate enough to locate the/a source of black powder formation, it should be possible to determine whether it was formed by MIC or chemistry and treated appropriately. MIC should be physically cleaned, treated with biocide, and consideration given to whether the pitting damage warrants repair or replacement. If possible, it would also be wise to look at modifying the pipe design to prevent the collection of water and the formation of biofilms.

Chemical formation of iron sulfide can be eliminated by removal or reduction of hydrogen sulfide or other gaseous sulfides from the gas. Although the resulting pipe wall thinning could be damaging over the long run, it is more generally distributed than MIC damage.

**Powder removal, handling.** Once black powder is generated in a pipe line, removing, handling and disposing of it is a necessary evil which must be dealt with until the material is prevented from forming. There are reasons to have concerns about how this is done. The black powder can be loose or adhered in the pipe, large or small quantities, dry like smoke, or wet and tar-like, possibly pyrophoric, and a nuisance or a hazard.

It is common for iron sulfide to form in a pipe and adhere to the surface in thin layers. The presence or amount of coating on the pipe may not be known in many cases, and is only revealed when the line is repeatedly pigged or washed. The presence of black powder in a pipe increases surface friction and roughness and increases the horsepower required to move an amount of gas through the pipe. Obviously these factors have negative economic implications.

The most common method for removing black powder from a pipe line is filtration at a compressor station. Removal at this point, however, has more to do with protecting the compressor than purely collection and removal of black powder.

Most iron sulfide tends to adhere to the metal where it forms. In some cases it is held in place by an encrustation of salt. Another factor that aids in holding the

powder in place is liquids. The powder adheres to wetted surfaces or collects in pools of water or hydrocarbon.

Factors affecting the movement of iron sulfide are dryness, changes in flow rate induced by pressure changes, and abrasive actions that shear the powder off pipe walls. This cleaning action is accomplished by abrasives moving through the pipe, water or other liquids, particularly in slugs, and pigging. Although all these mechanisms can happen incidentally as natural consequences of pipe line operation, they also are created intentionally to remove black powder buildup. Inspection pigging may be considered "incidental cleaning".

In some cases, mild abrasives, such as nut shells, are induced into a pipe section and retrieved downstream with the black powder they break loose. Such materials are most common in centrifugal compressor sites which can tolerate the materials passing through and cleaning blades and stators.

More commonly, liquids are used to wash down piping and remove iron sulfide. Although water is common and inexpensive, it is not good if any is left in the pipe to support MIC growth and oxidation. Diesel fuel or alcohol have been used as cleaning agents that will not promote microbial growth and are relatively inexpensive and disposable. Other cleaning agents are formulated by commercial suppliers specifically for cleaning pipe of black powder. These typically are more expensive than water, diesel fuel, or alcohol, but have fewer side effects. In most cases the liquid is trapped as a slug between two pigs. This causes it to fill the pipe more completely, control the liquid distribution, and provide better retrieval.

Pigging involves moving an object with scraping action through the pipe by gas pressure. The pig's cleaning action is to push loose material ahead of it out of the pipe as well as to scrape the walls of the pipe to remove more adhered material.

Pigging to remove black powder was discussed at the 1996 SGA Black Powder Summit. One of the more experienced companies indicated that using a heavy pig was more effective in breaking loose and moving black powder. Some indicated scraper pigs were effective, while others had less faith in their capabilities. It was noted that after several cleaning pigs were sent through a line, an intelligent pig passed through would then pick up much black powder. This is apparently due to

the fact that many iron sulfides are magnetic, and after being broken loose from the pipe wall into small pieces, the fine iron sulfide particles are attracted to the smart pig's magnetic field.

Pigs also are helpful in removing water and other liquids. The pig pushes the liquid down the pipe, sometimes causing it to combine with powder to form a sludge. This water removal is advantageous in preventing the growth of microbes.

Sections of pipe that cannot be pigged or otherwise opened for cleaning and inspection are candidates for MIC growth to the extent of wall penetration. Water that collects in such unpiggable sections or deadlegs is an even greater inducement to MIC and oxidation corrosion.

One difficulty with inserting anything, solid or liquid, into the pipe for cleaning is that the line must have been designed with cleaning in mind. Construction of deadlegs, side branches, and unpiggable pipe should be avoided from the standpoint of black powder formation. Even instrumentation line design should include a means for isolating and cleaning the line to prevent clogging and loss of signal.

None of the above methods should be construed as totally effective in the removal of iron sulfides from pipe walls. It is true that large amounts of material can be scooped from the bottom of the pipe, scraped from the walls, or washed loose, but corrosion engineers verify that it is very difficult to clean iron sulfides down to bare metal, even on steel pipe corrosion coupons. This requires a hydrochloric acid wash, subsequent neutralization with ammonia or bicarbonate, and then water wash to a neutral pH. This method requires a means to capture and dispose of released hydrogen sulfide. Such a process has been used in plant systems. Applying this method to a corroded transmission pipe is surely more difficult, and the steps must continue, to include removing any residual water as well.

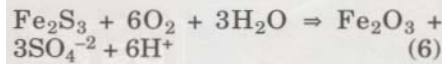
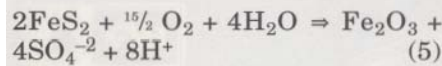
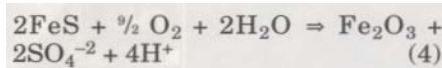
**Handling.** Once black powder is removed from pipe, whether by filter cleanout, pigging, or other access to pipe, compressor, or instrumentation internals, some attention must be

given to the safe handling of the black powder. The main hazard is associated with the pyrophoric nature of the material.

The greatest hazard seems to exist when the material is dry and in a fine powder form. Collecting the material in aggregates or chunks causes less combustion risk because the surface-to-mass ratio is smaller. Wet black powder, whether from water or hydrocarbons, is less likely to combust, because the liquid serves as a heat sink. General practice has been to wet down the powder with water as it is removed from pipe or filters to retard combustion. However, this method's drawback is that it is not permanent. When the material dries out, the risk of combustion returns. This may occur in the first depository after removal from the pipe, during transportation where it may be exposed to forced draft which induces drying and oxygen exposure, or it may not occur until the material is deposited into a landfill or other "permanent" site. There are several reports of black powder smoldering or catching fire in the backs of a trucks transporting it to a landfill. Others report that they store removed filters in a steel box and keep them there until they have smoldered and stopped, thus converting to iron oxide instead of iron sulfide, before taking them to a landfill. This tactic's downside is that sulfur dioxide is released when iron sulfide burns.

**Neutralizing iron sulfide.** There are several methods available for neutralizing iron sulfide. This investigation was able to identify a few of them. These methods involve actual chemical transformation of the material. The concern at this point is the nature of the chemical components produced by the reaction(s). It is possible to produce components less desirable than the iron sulfide.

The first method to neutralize iron sulfide is natural oxidation. The oxidation of iron sulfides is somewhat complicated. The reaction equations for the complete oxidation of FeS, FeS<sub>2</sub>, and Fe<sub>2</sub>S<sub>3</sub> by oxygen are given in Eq. 4-6.



Complete oxidation of iron sulfides in ambient conditions may only occur for FeS<sub>2</sub> (both pyrite and marcasite). Ferrous sulfide (FeS) is readily oxidized by moist air to form elemental sulfur and Fe<sub>3</sub>O<sub>4</sub>. At high temperatures, Fe<sub>2</sub>O<sub>3</sub> is formed. Ferric sulfide (Fe<sub>2</sub>S<sub>3</sub>) is readily oxidized to a hydrated ferric oxide and elemental sulfur. It decomposes at high temperatures to a mixture of FeS and FeS<sub>2</sub>. We also know that sulfur dioxide (SO<sub>2</sub>) was found after dust fires in pyrite mines. Oxidation of SO<sub>2</sub> continues in the atmosphere to produce sulfuric acid, resulting in acid rain.

A great number of reactions are possible in the black powder mixture of iron sulfides found in gas pipe lines, and the composition of products is going to depend highly on the material found in the black powder and on the reaction conditions. It is possible for a chemist to determine the equilibrium reaction products of iron sulfide compounds under different conditions. Sulfide compounds generally are stable only under very reducing conditions. This means that sulfide materials are usually only found in environments where there is a lot of organic material, such as in coal beds. This also explains their stability in natural gas pipe lines and their flammability in air. Reacting iron sulfide-containing black powder with an acid under reducing conditions will form hydrogen sulfide. Alternatively, a basic solution of a strong oxidizer may encourage rapid formation of sulfate before gas can evolve.

Potassium permanganate (KMnO<sub>4</sub>) is reportedly unique among oxidants that could be used to treat iron sulfide. It is safe to use and easy to apply and usually disposable. When applied as a less than 4% solution, it is relatively harmless if it contacts the skin, requires no special materials of construction, and does not form harmful or potentially explosive by-products.

The reaction of potassium permanganate (purple colored) and iron sulfide produces a brown liquid. When the iron sulfide is completely consumed, the excess permanganate solution again turns purple giving a positive visual indication when

the reaction is complete. The by-product of the reaction is manganese dioxide, which is biologically inert and can be discharged directly to a waste water facility. Depending upon the original contaminants, the solution may also contain oxidized organics or iron oxides. Reducing agents such as sodium thiosulfate or citric acid can be used if it is necessary to remove the manganese dioxide.

Another advantage of the potassium permanganate cleaning method is that it is faster than the multiple (more hazardous) steps of acidizing, thus reducing downtime. A pipe segment can be filled with the solution and either circulated with pumps or agitated with air, nitrogen, or steam. If the color of the solution turns brown, more permanganate should be added. If the color is purple, the reaction is complete and the segment can be drained and opened.

Potassium permanganate is more expensive than other potential oxidizing agents such as sodium hypochlorite, or hydrogen peroxide, or temporary treatment methods such as water saturation. Current permanganate costs are \$1.50 to \$1.80 per pound, while the alternative chemicals are about 30% less and water is considerably less. The cost advantage comes when compared against longer treatment times (downtime), waste handling and treatment, disposal problems and equipment, lack of undesirable side reactions, and worker safety.

**Disposal.** Almost all iron sulfide forms appear in nature, but in a pipe line it may mix with other liquid hydrocarbons to make the material a hazardous waste, or a material that must be deposited in a secure landfill. Since there are varieties of formulas for iron and sulfur combinations and a large number of other materials that could be combined with it. The classification of a hazardous material most likely must come from a test. The Toxic Characteristics Leaching Procedure (TCLP) test is designed to determine whether materials in the powder or sludge will leach into the soil to cause problems. This test must be performed on typical samples to determine whether any component has a hazardous flash point, is acidic

or corrosive, or is flammable. The pyrophoric nature of some forms of iron sulfide may make it a substance that requires controlled disposal.

#### ACKNOWLEDGMENT

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