Abstract

Black Powder is a typical contaminant in transmission pipelines. It is known for being detrimental to pipeline equipment and for causing operation and maintenance issues. Understanding its physical characteristics and its nature is necessary for pipeline operators in order to consider the appropriate separation technology, and to assess the possible root causes of its formation. An in-line sampling method is used to collect and to subsequently analyze the Black Powder transported in natural gas, condensate, or LPG. Recent site surveys in the Middle East confirmed the very fine size of solid particles and highlighted that particle size distribution varies significantly from one location to another. Different types of separation technologies exist to remove solid contaminants from pipelines, but their efficiency can be inadequate. The installation of a high-performance, properly designed filter, is an efficient and cost-effective solution to enhance the reliability of pipeline operation. Other separation technologies can also be considered to remove liquid water from pipelines, as a means to remove one source of the formation of Black Powder.

Introduction

Black Powder is a general term used to describe a host of corrosion related contaminants found in pipelines that transport natural gas, hydrocarbon condensates, and liquefied petroleum gas (LPG). It can also make its way from the pipeline to downstream processes damaging equipment through plugging and erosion. The composition of Black Powder has been found to vary significantly in chemical composition and to contain very fine particles down to sub-micron sizes. It can be a dry powder, a liquid suspension, or an intermediate sticky sludge depending on pipeline conditions. Previous studies have identified Black Powder to contain in some cases, primarily iron oxides$^{1,2}$ and in other instances, a mixture of iron oxides and iron sulfides$^{3,4}$. In gas streams containing carbon dioxide, iron carbonates can be formed as well. Also of importance is the presence of “binder” materials that may act in some instances to hold together smaller particles into agglomerates. These materials are typically of organic nature including paraffins, asphaltenes, and glycols. Additional non-corrosion related solids can also be present including sand or silt.

Black Powder is widespread to pipeline systems and is a global phenomenon. Early comprehensive studies of Black Powder have been carried out by the Gas Machinery Research Council$^{5,6}$ in the late 90's in the United States and subsequently papers on Black Powder have been published in a
Mechanism of Formation of Black Powder

Black Powder can be created by either chemical or microbial processes. Liquid water is a necessary condition for the reactions to occur. In addition to water, the presence of oxygen, hydrogen sulfide, and carbon dioxide have all been linked to creating favorable conditions for corrosion of the ferrous steel pipe. Natural gas that often meets stringent composition requirements with as low as 1 ppm of H\text{2}S can lead to high levels of corrosion.

Pigging operations in natural gas have documented extraordinary amounts of black powder ranging in 500 – 5,000 kg removed from scraped pipelines per run. Understanding the mechanisms behind the corrosion process is useful in coming up with preventative strategies.

Chemical Corrosion
Hydrogen sulfide will react with the iron found in the pipeline walls. Water will aid in the process and the presence of acid gases (H\text{2}S or CO\text{2}) will lower the pH of the water leading to iron dissolution, facilitating corrosion reactions. Details of the corrosion reactions have been covered previously with the main products formed being black pyrrhotite (Fe\text{1-x}S). In the absence of hydrogen sulfide, the pipeline will react with oxygen and water to form red-brown hematite “rust” (Fe\text{2}O\text{3}) or black magnetite (Fe\text{3}O\text{4}). A number of commercial inhibitors are available to reduce chemical corrosion. Reduction of oxygen, water, H\text{2}S, and CO\text{2} are also advisable as preventative measures.

Microbiologically Influenced Corrosion
In some instances sulfate reducing bacteria (SRB) can colonize a pipeline forming pockets on the pipeline wall where localized pitting corrosion can occur. These bacteria react with organic materials and sulfates to produce hydrogen sulfide and carbon dioxide. These bacterial products then go on to react with the iron in the pipeline walls creating different forms of iron sulfides. For SRB’s to exist, water is a key component to sustain life. Treatment options for reducing microbial influenced corrosion are to eliminate when possible the source of water and to dose with appropriate biocides.

Consequences of Black Powder
Black powder is known to consist of fine particles making it easy to transport inside pipeline systems. It is usually reported as being made of particles that shear easily into sub-micron size particles. Black Powder is also harder than the carbon steel used to make the pipelines and therefore poses an abrasive threat to erode the pipeline and pipeline components. The presence of Black Powder can have a large number of detrimental effects...
in the pipeline operations and also at customer’s delivery points including:

- Fouling of compressors – can require expensive maintenance as well as replacement parts. In worse case, it can cause catastrophic failure and loss of production.

- Blocking of orifice meters – When the orifice meters are compromised by contaminants, it is difficult to get an accurate flow rate and this can lead to losses in revenue to gas suppliers and affect downstream processes that are dependent on a flow rate.

- Contamination of instrumentation and control valves – fouling can lead to poor measurements, increased labor for cleaning, deterioration due to erosion, and to the need for replacement parts.

- Blocking of furnace nozzles – low NOₓ and Ultra-low NOₓ burners have been found to be more prone to fouling than earlier burner types. Burner design improvements to lower the NOₓ content have resulted in smaller orifices in the burner internals that make them susceptible to black powder contamination.

- Plugging filter systems – when excessive black powder is in the pipeline system, it can lead to short filter life creating increased labor requirements for change-out and added expense of replacement cartridges.

- Gas treating systems – if Black Powder is absorbed into the glycol dehydration or acid gas removal system it can cause a number of issues including foaming in the contactor towers and regenerators, resultant carryover of amine or glycol into downstream processes, off-spec gas, high maintenance on pumps, and excessive cost to replace contaminated solvents.

- Pipelines – Black Powder increases the interior pipe wall surface roughness. In some instances it can build up to sufficient levels on the interior pipeline walls creating a flow restriction. This then creates more pressure drop and requires additional horsepower to achieve the same flow rates and is an added operating expense. Severe or unchecked corrosion caused by Black Powder can ultimately cause the pipe to fail creating a safety hazard and major loss of production.

- Black powder disposal – when the black powder is made up of primarily pyrrhotite (Fe₁₋ₓS) this material exhibits pyrophoric properties and can auto-ignite often smoldering or burning when dried out. Special precautions need to be taken to dispose of spend filter cartridges and bulk solids.

- Pigging – the bulk removal of Black Powder is often accomplished prior to pipeline inspection by mechanical scraping of the interior pipe walls using pig devices. In some cases, chemical solvents or gels are used to make the pigging operation more efficient. This process results in associated labor, chemical, and waste disposal costs.

**Black Powder Diagnostics**

Understanding Black Powder’s characteristics is important to address the problem efficiently, particularly when the solution selected by the pipeline operator or the end-user involves filtration. In that perspective, it is important to establish the actual size of the particles transported in the pipeline and their distribution and quantity. Black Powder is usually reported as being in the sub-micron range but this does not take into account the possibility that small individual particles have agglomerated together, forming larger agglomerates. The evaluation of the presence of liquids (water, hydrocarbons, glycols, etc.)
is also of importance as it may aid in Black Powder formation, transport or create removal obstacles.

In diagnosing Black Powder issues in pipeline systems it is important to gain a comprehensive understanding of the problem. The composition of the gas or liquid stream transported is not sufficient. The following steps are advised:

1) Process Evaluation: Evaluate operations problems, pipeline history, data from pigging, gas composition (dew point, oxygen, acid gas content), and pipeline network features

2) Site Survey: Conduct Black Powder sampling and characterization, determine extent of liquid contamination, evaluate current separation devices

In conducting a site survey of contamination, the techniques used for sampling the contaminants deserve special attention, as well as the methods used to analyze the physical and chemical properties of the collected samples.

**Sampling Techniques:**

Previous studies\(^ {3,5} \) have focused on the laboratory analysis with less attention given to in-line sampling techniques. In most particle size evaluations, the Black Powder is collected from deposits in receiving traps or filter cartridges where it accumulated either by the pigging operations or by the filtration process. The sample is first dispersed in a solvent and sonicated into the smallest unit sizes that exist, typically in the sub-micron range with less consideration for how the particles may have actually agglomerated into larger sizes under operating conditions in the flowing stream.

When considering separation options for the removal of Black Powder, the actual agglomerated particle size distribution of the solid contaminants under operating conditions is important as the efficiency of most separators is dependent on particle size distribution. Therefore, representative samples of the solids from the stream should be collected using isokinetic probes that are inserted into the interior of the process pipe and match the velocities of the main process stream to the gas stream being sampled.

Both collection membranes and in-line particle counters can be used to sample the gas stream; however, particle counters will not be able to distinguish between liquid aerosols and solid particulates. Often total suspended solids are estimated using an equivalent spherical diameter and one particle density with particle counters, and this can lead to significant inaccuracy as Black Powder is known to have irregular shapes. Therefore, the technique of membrane sampling is preferable and as it collects contaminant, it also has the advantage of allowing for further chemical analysis of this material.

The use of isokinetic sampling combined with membranes allows for the collection of particles onto a sample membrane that will retain the same characteristic sizes as present under operating conditions. An apparatus for collecting solids from gases is presented in Figure 1.
Here a 47 mm diameter membrane rated at 0.1 micron in gas service is used to collect the Black Powder. The equipment contains a probe that is inserted into the process pipeline at the appropriate location and has a flow meter and control valve to adjust the flow rate to maintain the same kinetic energy (flow rate) of the gas in the sampling apparatus as the main pipeline. The probe can be inserted at different lengths to sample the gas at different locations within the diameter of the pipe (near the walls or in the center) and contains an internal hydraulic system that is powered by the pipeline gas pressure. Isokinetic sampling offers the best practice to remove representative particles as they are in the gas stream under operating conditions. A photograph of the Isokinetic probe is presented in Figure 2.

Two types of measurements can be made using this test equipment. A longer run is made to collect sufficient solids that are subsequently weighed to determine a total suspended solids content that is related to the total amount of gas sampled as measured by the flow meter. A shorter run is also made to collect particles that can later be analyzed in the lab for particle size distribution. This can be accomplished either manually using a light microscope to categorize the particles by size or automatically in the Energy Dispersive X-ray probe of a Scanning Electron Microscope (EDX-SEM).

As this procedure only provides a snapshot of the contaminant levels in the gas stream, it is recommended that these tests be repeated a number of times to see if the solids levels fluctuate. Similar equipment can also be used to sample liquid hydrocarbons including light condensates and LPG for particulates.

When evaluating pipeline corrosion issues, it is also important to determine if there are liquids present. When liquids are found, it is advisable to separate them as they are detrimental to pipeline operations creating flow restrictions. The presence of liquid water is also particularly important to know as it promotes corrosion and should be removed wherever possible. The pilot equipment to evaluate how much liquids are present in a gas pipeline is presented on the next page in Figure 3.

Here, a small scale high-efficiency liquid/gas coalescer is used to isokinetically sample a side stream taken off of the main operating pipeline. In this apparatus, the flow rate is measured downstream of the test coalescer using an orifice meter. The test is typically run over a period of a few days to gather data over a large enough time period to be representative. By emptying the collection sump at regular intervals, the amount of liquids in the gas stream can be charted over the test duration. This allows for analysis of any liquid content fluctuations and provides a broad based average rather than a quick snapshot of the liquid levels. Collection of liquid samples also allows for further characterization of the liquid contaminants.

Other techniques such as in-line particle counters can be applied to determine the liquid content and size distribution of the liquid aerosols, but these cannot distinguish between liquid aerosols and solid particulates and they don’t allow for the collection of liquids.
Recent surveys were performed in the Middle East on natural gas streams. The results indicated that the Black Powder particles had varying particle size distributions specific to each location.

### Export Sour Natural Gas Transmission Line, Middle East
Black Powder deposits were found in incoming gas and in the glycol dehydration system causing blocking of control valves and drain lines. The treated export gas was found to contain significant levels of solid particulates which caused complaints from the end user plants.

Two plant locations were selected for contaminant evaluation with both streams operating at 840 psig. Site 1 had a flow rate of 75 MMSCFD and a pipe diameter of 10
inches, while Site 2 had a flow rate of 130 MMSCFD and a pipe diameter of 12 inches. The average PSD and TSS data for the two sites are presented above.

Total Suspended Solids (TSS) contents on export lines at Sites 1 and 2 were 8.4 ppmw (235 g/MMSCF) and 8.9 ppmw (250 g/MMSCF), respectively. Iron sulfide appeared to be the main compound in Black Powder. Liquids were also determined using the liquid/gas coalescer pilot test equipment. Both water and hydrocarbon condensate were found with total liquid concentrations ranging from 0.4% to as high as 3.4%.

Note: the membranes used in preparing Figures 4-5 were not used for particle size distribution analysis, as the solids concentration was very high with overlapping particles.

Sales Gas Pipeline, Middle East
The presence of Black Powder in the sales gas network was found to damage control valves and to affect flow measurement at metering stations.

Two sites were sampled: both had pipeline diameters of 30 inches and pressures of 960 psig. Site 1 operated at 475 MMSCD and site 2 at 576 MMSCFD. The average PSD data for the two sites is presented on the next page in Table 2.
Black Powder Separation Options

The best strategy to deal with Black Powder is a long-term preventative program that addresses the root causes of its formation. It can involve a number of steps including the prevention of corrosion precursors, such as moisture or the use of internal coatings. Removal methods, however, provide immediate means to eliminate the Black Powder from flowing or building up inside the pipeline. The removal of contaminants adhering to the walls of the pipeline can be done by mechanical cleaning using pigs, or by chemical cleaning. These methods however do not prevent Black Powder particles from flowing inside the pipeline, which requires the use of separation technologies. While separation technologies do not address the root cause formation of Black Powder, and represent a cost for replacements and disposal (as other removal methods do), they are often the only possible solution to protect the pipeline components.

There is a variety of separation technologies currently used to separate Black Powder particles from natural gas or liquid hydrocarbon streams. Filters, filter-separators, and cyclone separators are the most common ones for gas pipelines. It is commonly reported, however, that particles flow through separation equipment and continue to cause problems in downstream equipment. The reason is that these technologies have variable removal performances which make some of them inappropriate to separate fine Black Powder particles. Variations in separator performance can be attributed to the device principles and separation mechanisms, the attributes of the filter medium and how it is designed, and to whether a separation device was adequately sized for the given application. Therefore, a good understanding of the advantages and limitations of each separation technology is necessary to select the appropriate one, in accordance with the actual characteristics of the contamination (size and quantity).

Solids Removal:

Some of the separation devices currently used for removing solids from gas include:

- **Gas Particle Filter** – consists of cylindrical cartridge filters that are placed in a vessel and typically start with a low differential pressure (DP) of a few psi. As the solids are collected, the filters have an increasing DP until they reach a terminal value and then the used filters are disposed and new replacements are installed. A wide number of designs are available with most having media wrapped around a metal core. Different types include string wound, pleated media, cylindrical surface, and depth style.
media where the pore gradient is varied in the cylindrical filter matrix to optimize solids collection. More recent designs have eliminated the disposable core and use permanent supports as part of the vessel. A number of different filter materials are available including, cellulose, glass fiber, and polypropylene as the most common types. The micron removal ratings of cartridge filters typically vary between < 1 micron up to 100 microns, with the appropriate rating being selected according to quality requirements.

An important distinction exists between how filters are rated, and the terms “nominal” and “absolute” can give very different performance for a filter labeled with the same micron removal. Nominal filters are generally not as reliable as absolute filters and are not subject to the same quality control or performance standards. The consequence is that particles larger than the claimed removal rating pass through the filter. In some cases nominal filters can collect contaminants and then as the pressure drop increases, release the trapped materials back into the stream. Absolute filters are tested in using particle counters to measure efficiency based on a distinct particle size cut off and made to more stringent and demanding specifications. They are designed to not release any trapped contaminants up until the design terminal DP.

- **Filter-Separator** – usually horizontal vessels that have a two-stage separation. The first stage is a filtration process that typically uses nominal filters and the second stage typically has a vane pack designed for liquid aerosol removal.

- **Cyclone Separator** – cyclones rely on centrifugal forces to separate particles from the gas, based on the cyclonic motion given to the gas when entering the pressure vessel or the small cyclone tubes (“multicyclone scrubber”). Solids are continuously separated from the gas and collect in an accumulator beneath the cyclone vessel. Cyclone separators have the advantage of normally not requiring any replacement of internals, unless subject to plugging or severe erosion. Cyclones however cannot separate very fine particles. Solid removal efficiencies of multicyclone scrubbers typically vary between 5-10 microns. The spinning velocity of the gas is a strong governing factor of the removal performance, which makes many cyclones generate high pressure drops, typically 10-50 psi.

- **Cyclo-Filter** – a new filter design that combines the advantages of a cyclone and absolute filter. It is a two-stage separator consisting of a low velocity cyclonic section to separate the coarsest particles down to 5-10 microns, and an absolute-rated cartridge filter section to remove the finest particles down to the requested specification. The cyclonic section operates at low velocities in order to generate a low pressure drop, typically a few psi. The cyclo-filter is a high-performance and high-capacity separator, that is capable of handling high solid contents including solid slugs during pipeline scraping operations.

**Liquid Removal**

Liquids are another possible type of contaminants in pipeline systems. Separation technologies that remove liquids are different from those that remove solids and Black Powder. A brief overview is shown below:

**Knock-Out Drum:** – typically an open tank that is intended to catch slugs or large liquid drop sizes based on gravity settling. Generally it is good for liquid drops > 300 micron. Its efficiency will increase with reduced flow rate as this will increase residence time.

**Demister Pad** – uses a mesh pad that coalesces liquid aerosols and is typically rated for drops
A Middle East petrochemical company is receiving natural gas from the gas grid. Supply is 95 MMSCFD at an operating pressure of 425 psig. The plant uses the gas for various purposes: as a process gas and it must be compressed prior to further processing, and as a fuel gas to feed a gas turbine and burners. Prior to entering this equipment, the total natural gas flow was passing through a knock-out drum equipped with a demister pad to remove liquids, then through filters to remove solid contaminants. Each filter vessel was equipped with 27 filter cartridges with a 4˝ diameter. These were cylindrical surface type filter cartridges, with a removal rating of 10 microns nominal as per supplier data.

Since 2003 the plant had been reporting several operational and maintenance issues, mainly on the compressor, which were causing recurrent maintenance activities due to cleaning and repair. Eventually these issues appeared to be related to Black Powder contamination transported with the feed gas. The plant reported that the impeller of the compressor had broken once during operation, which had caused damages on the casing and other internal parts. The plant also reported that control valves were blocking, and heavy black deposits were found in the compressor. In the inlet Knock-out drum, significant quantities of black materials were collected (2000 kg in 2003 and 1600 kg in 2004), and the demister pad appeared to be severely damaged. Finally filters were fouling very frequently, on average every two weeks. Analyses of deposits revealed that contamination was mainly made of iron sulfide particles.

The plant concluded that existing nominal-rated 10 micron filters were not efficient enough to remove Black Powder properly. In 2006 the plant entered into discussions with filter suppliers to help them solve contamination issues of the feed gas. The plant initially wanted to retrofit the existing 10 micron filter with 1 micron rated cartridges, and to install a new 5 micron rated filter upstream as a pre-filtration stage. In order to minimize the investment cost, and to optimize the filtration sequence, the plant finally considered a single stage filtration system equipped with 1 micron absolute-rated ‘Coreless’ filter cartridges with a depth, graded pore structure, polypropylene made filter media. A filter vessel was supplied, equipped with 30 ‘Coreless’ filter cartridges. During the sizing process, attention was given to the flow distribution around the filter.
elements in order to get favorable conditions for the formation of a filter ‘cake’ around each cartridge, thus increasing the solids removal capacity of the filter and its service life.

Filters were put on stream at the end of 2006. The dirty filters were replaced in mid-2008 after 20 months of successful operation, with no maintenance requirement on the downstream compressor. In 2007, after six months of operation, performance tests were run at the request of the plant, in order to measure the solids content upstream and downstream of the filter. The same apparatus as described previously was used for the performance test. Tests confirmed the good performance of the filter, with an outlet solids content of <0.01 ppmw, while the inlet solids content was 0.61 ppmw at the time of testing. At the opening of the filter vessel in 2008, the filter elements were in very good condition. The Black Powder appeared to be dry. As expected the finest particles had been trapped within the depth of the filter media, while the dry Black Powder had formed a 3-10 mm thick ‘cake’ on the outer circumference along the cartridges, which represented an additional dirt removal capacity to the filter elements.

The modification of the filtration stage with a properly designed technology has enabled the plant to reduce its annual maintenance costs through a more efficient protection of the compressor, as well as its operating costs due to less frequent filter replacements. The average filtration cost with the new filter technology is less than $10 / 100 MMSCFD, representing a reduction of about 10 times of their previous filtration costs.

**Conclusion**

Black Powder can vary considerably in size distribution, quantity, and chemical composition. A survey of field tests run on different gas streams demonstrated that the total suspended solids and particles in the range of 1-5 micron varied significantly. Isokinetic sampling is recommended as the best practice for sample collection to avoid altering the particle sizes. In-line membrane and pilot coalescer tests were found to be effective ways to evaluate solid and liquid contaminants in natural gas pipelines.

Before installing separation technology, site surveys are recommended to evaluate the pipeline contaminant issues. This way, the site specific contaminant characteristics can be determined and used as a guideline for selecting the optimized separation system. A summary of the most common solid removal and liquid removal technologies was provided. The effect of particle size distribution is of particular importance when selecting and sizing separators. Depending on solids loading and particle size distribution, either absolute filters or cyclo-filters are recommended as effective means to remove Black Powder.
References


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