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Black Powder in Sales Gas Pipelines: Sources and Technical Recommendations

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Abstract

One of the most severe problems affecting the efficient operations of gas pipelines is corrosion caused by black powder. According to the literature, the primary source for the existence of black powder is condensed water. In this case study, the temperature (40˚C) of the sales gas is much higher than its dew point (9.24˚C). The water is therefore in vapor phase. It is then proposed to remove water vapor from the gas at the entrance of the plant using an adsorption process. The recommended technology is the Layered Bed Temperature-Swing Adsorption (LBTSA) with micro-channels with molecular sieve zeolite 4A and activated alumina as adsorbents. In the case of presence of aerosols that could condense water, it is suggested to utilize a RED (Rare Earth Drum) magnetic separator in order to remove black powder from the gaseous feed.

Keywords

Black Powder, Sales Gas Pipelines, Temperature Swing Adsorption, Magnetic Filtration

1. Introduction

1.1. Sources and Consequences

One of the most significant challenges affecting the efficient operations of gas pipelines is corrosion caused by black powder. Uniform corrosion, galvanic corrosion, pitting and crevice corrosion are some of the main types of wet corrosion. On the other hand, dry corrosion refers to the corrosion process in the absence of condensed water. In this case, oxidation, sulfidation, carburization and decarburization are some types of dry corrosion [1]. For an oxidative dry corrosion, the temperature is usually around 370˚C and even higher temperatures are necessary for the other types of dry corrosion [1].

Fine particles of iron oxides, sulfides, and carbonates are the main components of black powder [2] [3] [4]. Black powder is harder than the typical carbon steel usually utilized for gas pipelines. This characteristic of black powder can accelerate further the erosion process. As a result, this erosion of the pipe wall speeds up accumulation of contaminants. Corrosion problems caused by the presence of these elements could be the source of up to 40% of pipeline malfunctions [5]. Understanding the theory and mechanisms of this type of corrosion is therefore fundamental to solve the problem from the source. Theoretically, the following three conditions are needed for corrosion to occur: a) two different metals or the same metal with two sides: one acts as anode and the other as cathode; b) an electrolyte; c) direct electrical contact between the cathode and anode. In this perspective, when dissolved in water, acid gases (H_2S, CO_2) and O_2 present in the gaseous hydrocarbons become highly corrosive and the different components of the black powder are formed by the below chemical and microbiological reactions:

a) Siderite-$FeCO_3$ is a product of CO_2 corrosion: dissolved CO_2 reacts with condensed water and the produced carbonic acid reacts with iron resulting in $FeCO_3$ formation [3].

$$\mathrm{H_2O + CO_2 \rightarrow H_2CO_3}$$

$$\mathrm{H_2CO_3 + Fe \rightarrow FeCO_3 + H_2}$$

b) Iron oxides are the result of two reactions: iron oxidation and the microbiologically induced corrosion (MIC). First, the iron oxide is present as α, β and γ forms. Due to the instability of γ type, it rapidly transforms to magnetite-$FeCO_3$. The following reactions describe the mechanism for the formation of magnetite-$FeCO_3$ [4]:

$$\mathrm{2Fe + H_2O + (3/2)O_2 \rightarrow 2 - FeO(OH)}$$

$$8\gamma - \mathrm{FeO(OH) + Fe \rightarrow 3Fe_3O_4 + 4H_2O}$$

$$\mathrm{FeCO_3 + (1/6)\ O_2 \rightarrow 1/3\,Fe_3O_4 + CO_2}$$

$$\mathrm{2Fe_9S_8 + 9H_2O + (27/2)\ O_2 \rightarrow 18}\gamma - \mathrm{FeO(OH) + 2S_8}$$

c) Iron sulfide is the product of the reaction between iron and hydrogen sulfide: $\mathrm{H_2S + Fe \rightarrow FeS + H_2}$.

As the concentration of H_2S increases, FeS_2 may be formed following the reaction [6]: $\mathrm{2H_2S + Fe \rightarrow FeS_2 + 2H_2}$.

In addition to water or moisture acting as the electrolyte, it is also a nutritious media for the SRB (Sulfate-Reducing Bacteria) cultures that grow and flourish in the gas pipelines. The reaction $\left(\mathrm{2H^+ + SO_4^{2-} + CH_4 \rightarrow H_2S + CO_2 + 2H_2O}\right)$ is an example of how SRB enhances the production hydrogen sulfide [2].

Black powder contains a variety of iron and sulfur compounds with different particles sizes and forms [7]. It can be a dry powder, a liquid suspension, or an intermediate sticky sludge depending on pipeline conditions. As shown in **Figure 1**, dry powder can be easily transported by gases which can affect the quality

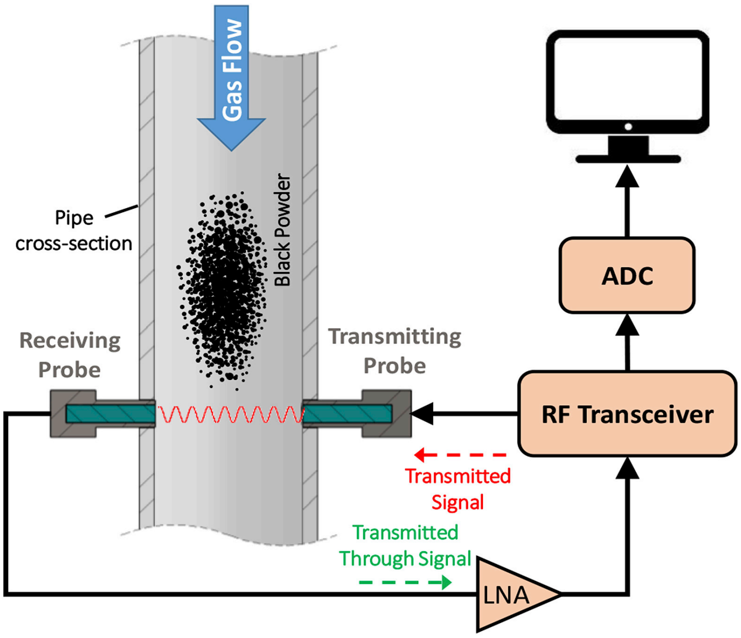

Figure 1. Black powder in gas pipeline [8].

of the product [2] [4]. On the other hand, if the substance has a tar-like appearance, it is commonly called wet powder. As indicated in **Figure 2**, wet powder tends to accumulate in the pipelines which could cause flow fluctuations as well as pressure drop [2] [4]. Moreover, plant components such as compressors, valves and furnaces can be damaged by abrasive wear or blocked by buildup. This can cause inefficient plant operations, intensive cleaning or expensive parts replacement.

1.2. Conventional Separation Techniques

The removal of the products of corrosion from the walls of the pipeline can be completed by mechanical cleaning using pigs, or by chemical cleaning. The most utilized cleaning methods are: a) Dry pigging: form of mechanical removal that can be done routinely on pig-gable lines and relies on the use of mechanical pigs running through the pipeline which does not involve taking line off-stream or introducing water to the system [4]; b) Gel pigging: in this process, gels can hold heavy deposits in suspension for days are pumped between mechanical pigs, which remove the debris from the pipe wall, distribute it through the volume of the gelled fluid, and contain the gel to give 360-degree coverage [4].

On the other hand, separation techniques prevent black particles from flowing inside the pipelines and damage the downstream equipment of the plant through plugging and erosion. In this perspective, there are a variety of separation technologies currently used to separate black powder particles from natural gas or liquid hydrocarbon streams. For example, filters, filter-separators and cyclone separators are the most common separation techniques for gas streams: a) Gas Particle Filter: consists of cylindrical cartridge filters placed in a vessel and

Figure 2. Wet black powder [9].

typically start with a low differential pressure of a few psi; b) Cyclo Filter: is a new filter design that combines the advantages of a cyclone and absolute filter [2]. However, one of the most serious problems affecting performance of units and equipment in the gas industry is the presence of micro particles that cannot be filtered by conventional way. Conventional filtration systems are not able to retain micro particles carried out by the gas. The efficiency of filtration could be increased by decreasing the size of the pore of the filter. However, it will also decrease the flow rate of the gas to be filtered. Moreover, the filter cake grows in the course of filtration, becoming "thicker" as black powder particles are retained. As a result, the gas flow rate decreases due to blinding and back pressure.

1.3. Magnetic Separation Techniques

All materials can be classified based on their magnetic properties. Minerals separation based on magnetic susceptibility is accomplished at various intensities and in different basic machine configurations. "Paramagnetic" minerals are attracted along the lines of magnetic force to points of greater field intensity. "Diamagnetic" minerals are repelled along the lines of magnetic force to a point of lesser field intensity. "Ferromagnetic" minerals, a special category of paramagnetic materials, have a very high susceptibility to magnetic forces and retain some magnetism (remnant magnetism) after removal from the magnetic field [10]. **Table 1** shows the common ferromagnetic and paramagnetic minerals as well as the field intensity that is required in order to separate those minerals.

Magnetic Separation is the process of separating components of mixtures by using magnets to attract magnetic materials. According to the different sorting principle of magnetic separator, it can be divided into cross-belt magnetic separator (CBMS), induction roller magnetic separator (IMRS), lift roller magnetic separator (LRMS), permanent magnet roller magnetic separator (RERMS), Rare earth drum magnetic separator (RED) and High-intensity rare-earth roll (RER) separators [12]. Recent improvements in magnet composition and design have led to the development of dry permanent magnetic separators. These improved rare-earth permanent magnets (e.g., NdFeB magnets) have a magnetic attractive

Table 1. Common ferromagnetic and paramagnetic minerals [11].

	Mineral	Formula	Field Strength (kGauss)
Ferromagnetic	**Magnetic**	**Fe_3O_4 (Black powder)**	1
	Pyrrhotite	**Fe_7S_8 (Black powder)**	0.5 - 4
Paramagnetic	Ilmenite	$FeTiO_3$	8 - 16
	Siderite	**$FeCO_3$ (Black powder)**	9 - 18
	Chromite	$FeCr_2O_4$	10 - 16
	Hermatite	**Fe_2O_3 (Black powder)**	12 - 18
	Wolframite	$(Fe, Mn)WO_4$	12 - 18
	Tourmaline	$Al_6B_3Fe_3H_{10}NaO_{31}Si_6$	16 - 20

force an order of magnitude greater than that of conventional permanent magnetic circuits. The two main types of dry permanent magnetic separators that have found wide industrial applications are the rare-earth drum (RED) and the rare-earth roll (RER) magnetic separators [10].

1.4. Gas Adsorption Systems

Existing industrial purification plants are based on absorption, adsorption and Joule-Thompson effect (JT effect) techniques. In general, Absorption is used in cases when emphasis is not placed on the water content of the output stream, and when low operating and capital investment are required. Adsorption is used in cases when bone dry NG is required. Low temperature separation employing the JT effect is used in cases where a sufficient pressure drop is available between the input and the output of the dehydration unit.

Gas adsorption can be categorized as a separation where a small amount of an impurity is removed from the gas stream. The adsorption system is typically a fixed-bed where the process occurs in a specific zone in the bed called mass transfer zone (MTZ). The adsorbents become saturated when the rate of adsorption and desorption become equal. At this stage, the exhaustion point is reached and the bed needs to be regenerated (**Figure 3**) for the next adsorption cycle.

The two most common regeneration methods proposed in the literature are: a) rise in temperature by TSA (Temperature-Swing Adsorption) and b) decrease of partial pressure by PSA (Pressure-Swing Adsorption). Displacement with stronger adsorbing species, purging with an inert fluid and changing of chemical conditions are some other technical methods of regeneration. **Table 2** shows the benefits and disadvantages of some important desorption methods.

2. Literature Review

Black powder is a persistent and extensive type of contamination. According to the literature, many gas plants have experienced technical problems related to black powder. For example, 1000 kg of black powder accumulated in the first 12 km of a Greek pipeline [6]. Moreover, in Houston (USA) around 3600 kg of

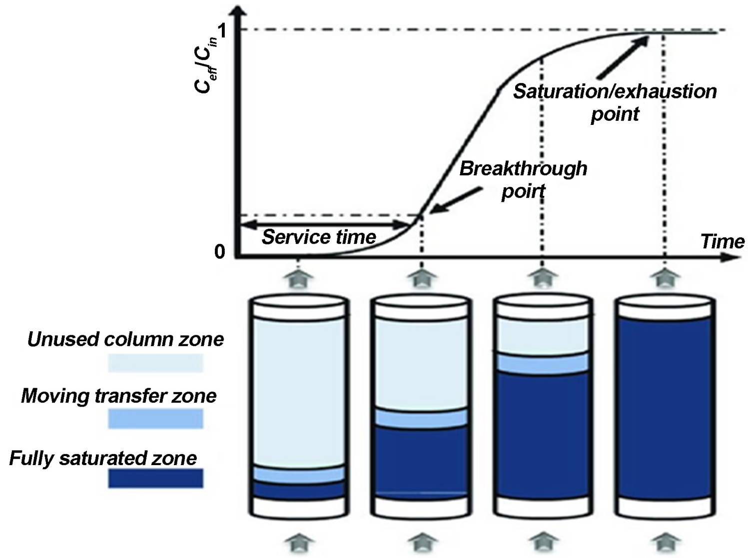

Figure 3. Progress of mass transfer zone (MTZ) [13].

Table 2. Advantages and disadvantages of different dehydration methods [14].

Regeneration method	Advantages	Disadvantages	Utilization
Temperature-Swing Adsorption (TSA)	1) Applicable for strongly adsorbed species 2) High concentrations recovery for gases and liquids desorbate	1) Thermal aging of adsorbents. 2) Thermal inefficiency due to Heat loss 3) Inefficient use of adsorbent if the cycle time is long 4) High latent heat for liquids 5) Low concentrations	Drying gases Drying solvents
Pressure-Swing Adsorption (PSA)	Good for weakly adsorbed species required in high purity	1) Very low pressure may be required 2) High concentrations	Drying of gases Hydrogen recovery Air separation
Adsorbent Displacement	1) Good for strongly held species 2) Avoids risk of cracking reactions during regeneration 3) Avoids thermal aging of adsorbent	Product separation and recovery needed	Separation of linear form branched and cyclic paraffins
Inert Purging	Essentially at constant T and P	Only for weakly adsorbed species, purge flow is high not normally used when desorbate needs to be recovered	Relatively uncommon without TSA since purging alone is only suitable for weakly adsorbed species

black powder was removed from a gas pipeline [2]. More significantly, in extreme situations, it can result in completely stopping sales gas supply to customers. For example, a single blackout incident, for a duration of three days, costs over a $1-million, Abu Dhabi National Oil Company (ADNOC) estimates [8].

To successfully remove black powder in pipelines, a deep understanding of its sources, formation process and material characteristics is needed. In this perspective, significant research efforts have been dedicated to solve this technical concern. While some researchers have focused on the thermodynamic aspect, others have centered their attention on the chemical and physical analysis of the black powder. For example, a number of analytical techniques such as Micro-hardness, nano-indentation, X-ray fluorescence (XRF), scanning electron microscopy (SEM), X-ray diffraction (XRD) and energy dispersive spectroscopy (EDS)were performed on various samples of black powder in order to identify the relationship between corrosion initiators (H_2S, H_2O, O_2, & CO_2) and the components of the black powder. For example, it has been shown that the accumulation of black powder is caused by the black pyrrhotite that is produced when water, oxygen, hydrogen sulphide and carbon dioxide react with the ferrous steel pipe.

Moreover, a number of studies suggest that iron sulfide could be the major component of black powder [3] [6] [9]. Some researchers have investigated the sources of black powder and the ways for mitigation. One study claims that H_2S is the major contributor to black powder formation. The analysis shows that 1 ppm of hydrogen sulfide in the pipeline with a flow rate of 10 MMCFD could result in the annual production of 363 kg of black powder (iron sulfide) [9]. The authors also affirm that iron oxides and other contaminants give the powder its black color [9]. Other reports indicate that, depending on the source of gas, iron sulfide could be a major component if the powder formed within sour gas or a minor component when formed in the sweet sales gas [2].

Analyzing the components of black powder, it was found that Fe_3O_4, α-FeOOH and γ-FeOOH are the major components of black powder of the sweet sales gas. On the other hand, $FeCO_3$ was identified as a minor component. However, no iron sulfur-based compounds were identified [2]. Regarding the components of the sour gas black powder, the major components were FeS and FeS_2 and the minor components were Fe_3O_4, α-FeOOH, γ-FeOOH and $FeCO_3$ [4]. Furthermore, it is demonstrated that even if the moisture level in the sweet sale gas pipeline is as low as 0.12 to 0.55 mg/L, it is considered to be sufficient for causing internal corrosion independently of the other components of the gas [2]. To summarize the results of the literature, **Table 3** indicates the necessary conditions and the composition of the resulting black powder. From **Table 3**, it can be observed that condensed water is the needed electrolyte for any component of black powder to appear and therefore, the source for the corrosion in gas pipelines.

According to the literature, the size of black powder's particles is from above 500 microns to well below 1 micron. Often, samples show high particle concentrations below 10 microns in size, exceeding 50% of the total contamination. Further, up to 50% of black powder contamination can be at 5 microns or less; 30% of it can be at 1 micron or less [15]. Sherik reported 50% of contamination of gas pipelines in Abu Dhabi was between 3 and 5 μm, while only 5% was in the 50 to 100 μm range [16]. Moreover, Mueller analyzed wet and dry samples in laboratory and the experimental results show that 81.6% of black powder samples had a size less than 1 μm [17].

Over the past 15 years, significant improvements in rare earth magnetic technology have created the ability to use magnetic fields as a filter or separator medium which is environmentally and user friendly, highly efficient and cost

Table 3. Components of black powder and its source [2] [3] [4] [6] [9].

	Source						
Black powder components	Condensed water	H_2S	CO_2	O_2	(Sulfur Reducing Bacteria)	(Acid Producing Bacteria)	(Iron Oxidizing Bacteria)
Siderite-$FeCO_3$	X		X				
Iron Oxides	X			X		X	X
Iron Sulfides	X	X			X		

effective [15]. Rare Earth Magnetic Separators (REMS) are now commercialized for the separation of minerals. The main advantage is that rare-earth magnetic filters can stop particles smaller than one micron in size while conventional filters cannot stop particles smaller than 5 - 10 microns [18]. Moreover, unlike conventional filtration, in magnetic filtration, the filter will not block so there is no blinding or pressure build up.

Filtration and separation techniques are corrective actions aiming to reduce the negative effects of black powder on the equipment of the plant. According to the literature, there will be no black powder in gas pipelines without the presence of condensed water in the gas stream. A long-term preventative program is therefore needed in order to address the root of the problem. In this perspective, the main objective of this study is to select the suitable technology that could prevent the formation of black powder, which means eliminating water from the gas.

3. Case Study of Black Powder

The gas plant under investigation has a major technical problem linked to the formation of black powder in one of the sales gas pipelines. The annual collected amount of black powder was estimated to be around 750 kg. The objective of this study is to find technically feasible solutions that could prevent black powder formation. In this perspective, the investigation includes the following steps.

3.1. Feed Gas Operating Conditions and Composition

The literature shows that there will be no black powder formation without condensed water in the gas stream. Thermodynamically, water is condensed if the temperature of the gas is below its dew point. Information about the dew point of the gas transported is therefore fundamental for the determination of the phase of water in the pipeline. For the gas under consideration, the operating conditions of the pipeline and the composition of the gas are shown in **Table 4**.

Based on the parameters shown in **Table 4**, the probability to have condensation of water is very small because of the low dew point. The water in the pipeline is therefore assumed to be in the vapor phase. The most recommended technology in this case study is the fixed bed adsorption using advanced hydrophilic adsorbents.

Table 4. Feed gas operating conditions and composition.

Operating conditions and composition of the gas	
Flow	1000 (MMSCFD)
Pressure	41 barg.
Temperature	40˚C
Dew Point	9.24˚C
Methane	90 (% mol.)
Ethane	5 (% mol.)
Propane	1 (% mol.)
C_4^+	1 (% mol.)
N_2	1(% mol.)
CO_2	2 (% mol.)
H_2O	150 ppm
H_2S	20 ppm

3.2. Main Recommendation

Adsorption is a very used separation process that has wide industrial applications such as gas purification, liquid purification, ion exchange and chromatography. Since the operating conditions suggest the existence of water vapor at very low concentration, the gas could be dehydrated at the entrance of the gas plant using an adsorption process. As extensively described in the literature, the two most common adsorption techniques are the Temperature Swing Adsorption (TSA) and the Pressure-Swing Adsorption (PSA).

3.2.1. Selected Technology

Recently, the feasibility of the use of adsorbent-coated microchannels in temperature swing adsorption (TSA) processes for natural gas purification has been investigated by analyzing fluid flow and heat and mass transfer within the monolithic structure containing microchannels [19] [20] [21]. The objective was to overcome the operational problems due to heating usually utilized in adsorbent beds for the TSA process. These technical issues have been minimized by introducing the adsorbent as a thin layer coating the microchannel walls. As a result of the new design, the overall performance of the TSA-based purification process was found to be competitive with existing PSA-based cycles, yielding similar or higher product purities and recoveries and at least an order of magnitude greater purification capacity [19]. Based on this new technology and the data available in **Table 3**, it can be concluded that TSA with microchannels is the most suitable adsorption process in this investigation because a) water is a strongly adsorbed element and b) the concentration of water in the gas is very low (150 ppm).

3.2.2. Selected Adsorbent Type

Many adsorbents have been introduced in the literature for the adsorption process such as silica gel, molecular sieve carbon, activated alumina, activated carbon and molecular sieve zeolites. The effectiveness of zeolites for water removal is based on three important characteristics: a) the channel diameter acting as a filter, which limits the number of species that can co-adsorb; b) the polar environment created in the channels, which creates an environment in which preferentially polar molecules are adsorbed and c) potential for adsorbing and desorbing water without causing any damage to the crystal structure [22] [23]. Moreover, when deep removal is required (below 1 ppmv), molecular sieves are preferred.

The basic formula for zeolite molecular sieves is $M_{x/m}\ [(AlO_2)_x(SiO_2)_y]\ z\ H_2O$ where M is the cation with valence m, z is the number of water molecules in each unit cell, and x and y are integers such that y/x is greater than or equal to unity. Depending on the pore size and cations attached, several types of zeolites are available in the market [24]. The 4A molecular sieve is an alkali metal aluminosilicate with an effective pore opening of approximately 4 angstroms. The sodium form of type A is widely used as a general-purpose drying agent and has good physical and adsorption properties. Type 4A beads can be used to adsorb water, ammonia, methanol, ethanol and carbon dioxide [25].

Finally, the LBTSA (Layered Bed Temperature-Swing Adsorption) processes are an upgrade of the TSA method. Here, the adsorption column is composed of several layers of different adsorbents. Hence the properties of the separate adsorbents are combined in a single column. For example, in NG (Natural Gas) dehydration a combination of activated alumina with molecular sieve 4A is used. Alumina has better resistance to liquid water, so a thin layer is put in first place to contact the wet NG. This ordering supports the lifetime of the molecular sieve, which is placed below the alumina layer [26].

3.2.3. Description of the TSA's Operation Process

The proposed process scheme of the adsorption unit using the TSA system for regeneration is illustrated in **Figure 4**, where two vessels are operated cyclically. One vessel undergoes adsorption while the other goes through regeneration.

First, temperature is a very important process variable for any TSA adsorption unit. Temperature control of the adsorption and desorption columns is therefore vital to ensure efficiency of both processes. In this perspective, three recommendations need to be respected: a) during adsorption: the temperature should be high enough for the adsorbent to release the water content; b) during desorption: ensure that the new elevation temperature will not cause the degradation of the adsorbent and c) full utilization for the bed capacity requires that the adsorption's time to be slightly higher than the desorption's time.

During normal operation, effluents of the adsorption vessel are sent to the storage from where it is sent to consumers. A slipstream from the effluents is utilized in regeneration. The effluents are heated using a fired heater to the experimentally determined regeneration temperature then passed through the vessel undergoing regeneration. The outlet gas from the regeneration step is used as

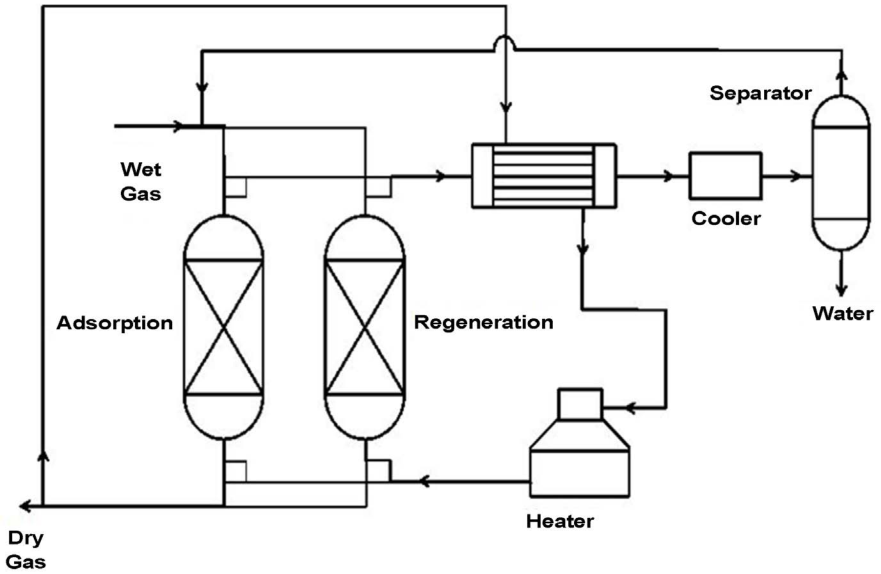

Figure 4. Temperature swing adsorption process [26].

fuel gas. After completion of the regeneration cycle, outlet effluent is used directly for the purpose of cooling the vessel down to the adsorption temperature. Once the vessel has reached the normal operating temperature, control valve positions are switched to introduce the feed gas to the regenerated beds while introducing the regeneration gas to the adsorbed beds. The amount of the slipstream is around 5% - 10% from the effluents.

3.3. Second Recommendation

Aerosols are very small particles of solid or liquid suspended in the gas. The water aerosols have different sizes depending on the air conditions. In the clean air, the diameter of aerosols was found to be <3 μm [27] whereas in polluted air, the aerosol's diameter was found to be <1 μm [27]. Because of the presence of these aerosols, condensation of water could take place even if the gas temperature (40˚C) is higher than the dew point (9.24˚C). In this situation, condensation of water could happen because of the surface tension. Such heterogeneous nucleation has been well described by thermodynamic laws such as the Kelvin equation [28]. In this case, aerosols initiated the formation of black powder. In this case, black powder will be filtered using a REMS (rare-earth magnetic separator) at the entrance of the plant.

As shown in **Table 4**, because of the relatively high concentration of carbon dioxide (2% mol.) in the gas stream, Siderite-$FeCO_3$ is expected to be the major product of the black powder collected in the gas plant. On the other hand, due to the very low concentration of H_2S (20 ppm) in the gas, Iron sulfide could be a minor product of the collected black powder formed by the reaction between the iron pipes with hydrogen sulfide. Based on the literature review, REDMS (Rare earth drum magnetic separator) is the most suitable magnetic separation technology. The strength of the magnetic field will depend on the exact composition of the black powder. For Siderite ($FeCO_3$), a magnetic strength up to 18 kG is needed (Table 1).

4. Conclusion

Condensed water is described in the literature as the primary source for the existence of black powder in gas pipelines. The proposed recommendations are based upon two assumptions. First, when the operating temperature is higher than the dew point of the gas, there is no condensation of water. Therefore, water vapor could be eliminated from gaseous feed. To reach this goal, Layered Bed Temperature-Swing Adsorption (LBTSA) with microchannels was proposed with molecular sieve zeolite 4A and activated alumina as adsorbents. In this case, the acid gases H_2S and CO_2 will also be adsorbed. Secondly, for the case of the presence of condensed water, it is recommended to filter the black powder by a RED (Rare earth drum magnetic separator) magnetic filtration.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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