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A Study of Chemical Cleaning to Remove Deposits in a Cold Box: The 10th Refinery of South Pars

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ABSTRACT

The 10th Refinery of the South Pars Gas Complex in Iran experienced a notable decrease in gas production due to the formation of deposits in the cold box. To tackle this issue, a comparison of data was conducted before and after the refinery's annual turnaround. After extensive investigations, it was determined that the Xylene solvent wash was the most suitable solution to address the cold box pressure drop in the ethane recovery unit. The scale of the cold box studied in this article is significant in terms of dimensions and flow rate. The Phase 19 gas plant encountered the issue in interesting way according to limitations such as large-scale continuous production. A chemical wash procedure was designed and implemented during the refinery shutdown, which resulted in a successful reduction in the cold box pressure drop and an increase in production rate by approximately 188 tons/hour. This led to a yearly revenue increase of approximately 387 million USD, emphasizing the significance of addressing deposit buildup in cold boxes to maintain optimal gas production and achieve substantial financial gains.

1. Introduction

Nowadays Natural gas is a strategic energy source that widely used all over the world. It has many advantages rather than other fossil fuels encourages increasing its world consumption and expected growing demand [1]. So, it is vital to reduce the production cost and improve the efficiency of refining processes by maximizing the capacity of current processes and process troubleshooting. Production gas capacity may decrease by many factors as number of researchers mentioned it [2, 3].

Increasing the pressure drop of the unit is one of the causes of production capacity reduction. Pressure drop constraint is mainly factor in gas plants that determines how much flowrate allows to pass through the production process. Despite of upstream and downstream condition problems which lead to decrease production, fouling is mainly cause of increasing pressure drop and decreasing production capacity.

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According to Speight [4], fouling is deposit formation, encrustation, deposition, scaling, scale formation, slagging, and sludge formation which has an adverse effect on operations. In other word fouling is the accumulation and formation of unwanted materials on the surfaces of processing equipment. Fouling is an extremely complex phenomenon. Fundamental fouling may be characterized as a combined, unsteady state, momentum, mass and heat transfer problem with chemical, solubility, corrosion and biological processes may also take place [5].

Fouling affects both capital and operating costs of processes and equipment such as heat exchangers. The presence of fouling in the surface of heat exchangers decreases the ability of the unit to transfer heat. The need to restore flow and heat exchanger efficiency will necessitate cleaning. Scheduled interruptions to production can help minimize losses, but even if the rest of the plant is functioning properly, it can still result in reduced output and profits. This, in turn, can affect the overall costs of the specific sites. The consequences of enforced shutdown due to the effects of fouling are of course much more expensive.

Fouling in petrochemical industries is a common issue that can cause significant reduction in production. The main factors affecting fouling include fluid flow velocity, fluid properties, surface temperature, surface geometry, surface material, surface roughness, and the concentration of suspended particles, solids, and impurities. The nature of the process determines the source and reasons for fouling, which can manifest as sludge, sediment, and darkening of the liquid. The sludge or sediment can increase the viscosity of the liquid, leading to fouling in the storage tanks or remaining in the liquid as an emulsion. The petrochemical industry often experiences fouling due to the viscous nature of their fluids, while natural gas processes may face fouling in the form of wax formation in cold sections and where two-phase hydrocarbon flow exists. Fouling in the cold box heat exchanger leads to a high pressure drop and can limit plant capacity, leading to a reduction in production rate.

The total capacity of South Pars gas plant Phase 19 onshore facilities is 2000 MMSCFD of dry reservoir fluid includes all processing units, offsites, and infrastructure necessary to produce sales gas, ethane gas at the required specification, commercial grade liquid propane and butane, solidified Sulphur and stabilized condensate. A total of 4 GTUs are provided, two for each phases 19 A/B. The gas treating units (GTUs) are processing sour gas from the inlet reception facilities of the plant where contains small amount of contaminants such as H_2S, CO_2 and organic Sulphur compounds (mainly mercaptans), etc. The sweetening unit utilize Methyl-Di-Ethanol-Amine (MDEA) technology to selectively remove most of the hydrogen Sulfide to the required specification 5 mg/sm3 and carbon Dioxide from the sour gas. The sweet gas subsequently routed to gas dehydration unit to remove water and Mercury, by means of four parallel and identical molecular sieve units where the Dew point is set up to -95°C at 29 barg (bar gauge) and water content to about 0.1 ppmv. Dry sweet gas passes through the after-dryer filters and mercury guard to reach the mercury content value of 10 ng/Nm^3. Since the ethane recovery unit uses cold box heat exchanger to make refrigeration that made of aluminum plates is essential to remove mercury to avoid aluminum destruction. Then the dry sweet gas feeding the ethane recovery unit, which is designed to draw off an ethane cut from it. Dry sweet gas is first precooled down to -35°C in cold box by exchanging heat with different cold streams. The outlet main gaseous stream is fed to expander that operates as driver of treated gas compressors provided to feed sales gas to export. The lean gas produced is first heated and then routed to compress for export. Ethane cut is first heated and then routed to ethane treatment unit for further treatment. NGL cut is directly routed for fractionation [6].

To better understating the issue its need to look briefly to ethane recovery unit with some details. After H_2S removal and H_2O and mercury removal, the dry sweet gas is routed to the ethane recovery unit where the gas is processed to

separate the following product streams: treated gas sent for export, gaseous ethane sent to ethane treatment unit for CO_2 removal and drying, propane and heavier compounds sent to NGL fractionation. Ethane recovery unit of each gas train comprises the following systems: feed gas cooling in a cold box unit and expansion in the feed gas expander, demethanizer and export gas, deethanizer (Fig. 1). Sweet and dry gas from the dehydration is first precooled down to -35°C in cold box by exchanging heat with different cold streams: A propane stream at medium pressure (Propane at 19.3°C); The cold sales gas coming from demethanizer column and cold box at -87.3°C and going to the gas/gas exchanger at 16.8°C; A withdrawal stream from and to lower section of demethanizer (entering and leaving cold box at 12.6°C and 18.5°C); A propane stream at low pressure (Propane at -4.3 °C); A withdrawal stream from and to medium section of demethanizer (entering and leaving cold box at -37.1°C and -11.9°C).

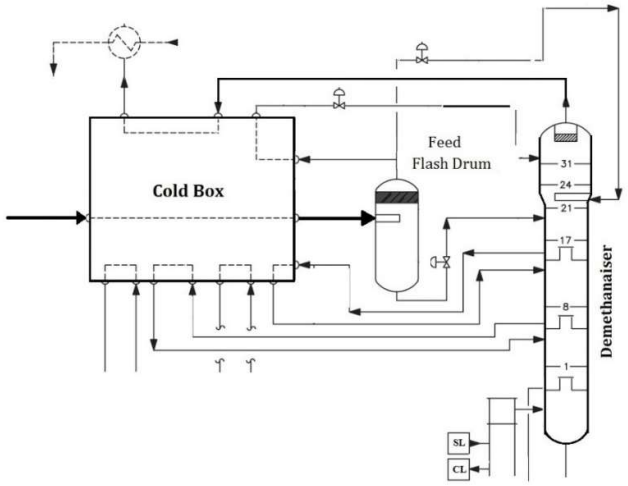
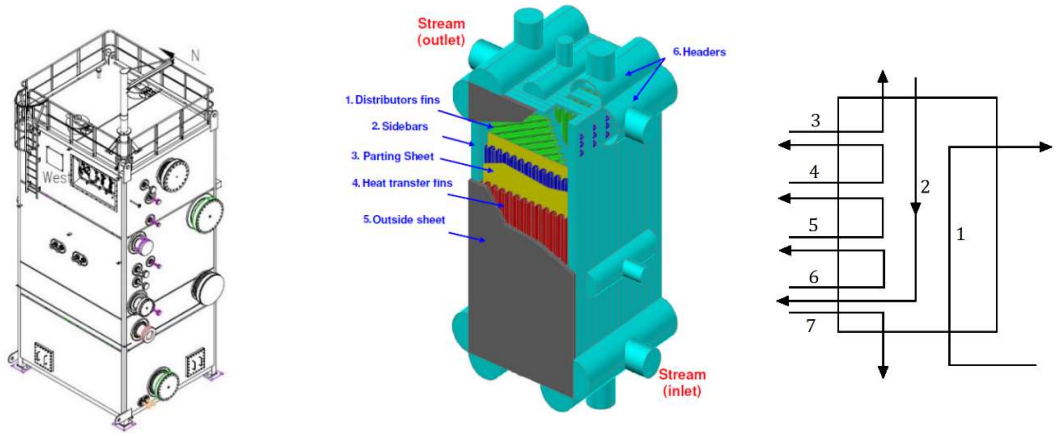


Fig. 1. Ethane recovery unit process flow diagram

A fives Cryo's brazed plate-fin heat exchanger is made of aluminum. It is generally of rectangular shape. Alternating layers of corrugated fin constitute a plate-fin heat exchanger. To prevent leaks of fluid, and to isolate exchanger to each other, these layers are separated between them by parting sheets. They are attached by means of side bars, and are provided with inlet and outlet ports for the streams. The assembly is brazed in a vacuum furnace to become a rigid core. Over openings there are header tanks welded to deliver and collect the fluid from the core. Customers can connect their pipeline to the plate-fin heat exchanger, thanks to a nozzle that is welded on the header tank (as Fig. 2(a), (b) and (c) illustrates).

At -35°C temperature a major portion of the butane and heavier components are condensed. The two-phase stream from the cold box, enters feed flash KO drum, where the two phases are separated: the liquid phase directly feeds the demethanizer column, while the gaseous phase is split into two streams. The main is sent to the feed gas expander, before entering the demethanizer column, the second flows through the cold box, before entering the column at the top as reflux stream. In the cold box, the second gaseous stream from feed flash drum is condensed at -84.5°C by exchanging heat with sales gas flowing from top of demethanizer at – 87.3°C; at cold box outlet condensed hydrocarbons are flashed through a flow control valve at 29 barg and -95.6°C and sent to the top of demethanizer as a column reflux, to improve C_2+ recovery efficiency and light products separation. Liquid stream from feed flash drum is flashed at 29.4 barg and -48.2°C through level control valve, (about 25% of flashed gas) and fed to demethanizer.

The primary gas stream from the feed flash drum is sent to an expander which drives the treated gas compressors to supply sales gas to the export unit. After expanding at 29.5 barg and -66.5°C, the gas stream is fed to the demethanizer column. This column separates on-spec sales gas from the top and wide-range gasoline from the bottom. Light components are stripped from the liquid phase in the column section below the feed tray, and heavier compounds are absorbed by the liquid reflux from the cold box in the column section above the feed tray. The fractionation process requires heat input from a LP steam reboiler. To reduce LP steam demand, two intermediate withdrawals are provided, both of which are reheated in the cold box package using cooling dry gas. The bottom product from the demethanizer column is sent to the deethanizer column, which removes ethane as an overhead vapor stream and yields a bottom product containing all the propane and heavier compounds.



(a) (b) (c)

Fig. 2. CRYOFIVE cold box heat exchanger: a) Cold box structure in the plant; b) Cold box configuration of heat exchanging sheets, fin and headers; c) Hot and cold streams passing through the cold box and stream No. corresponding their tags are as follows: 1) Sale gas; 2) Feed line; 3) Propane first stag; 4) Lower demethanizer side stream; 5) Propane second stage; 6) Upper demethanizer side stream; 7) Reflux to demethanizer.[11]

The scale of the cold box studied in this article is large and significant, both in terms of dimensions and the amount of flow of fluids that exchange heat with each other. From this point of view, one of the prominent features of the present work is providing valuable real industrial data for this equipment. The way the phase 19 gas plant encountered the cold box issue is also interesting according to its limitations such as large-scale continuous production without having a chance and opportunity to review the design of the cold box and only through the creation of minor modifications and operating instructions. However, there are valuable articles in this field that we will take a quick look to them here. Awais and Bhuiyan [7] investigated different ways of enhancing heat transfer rate and pressure loss reduction in various types of compact heat exchangers (CHXs). Their study was an attempt to provide designers innovative techniques of designing compact heat exchangers (CHXs) incorporating highly effective heat transfer performance, moderate pressure drops and less pumping power with low area to volume density and cost-effective products.

Wang and Yanzhong [8] mentioned that studies of plate-fin heat exchangers are currently focusing on four areas: heat transfer calculation, surface analysis, flow resistance and design optimization. They reviewed traditional design approaches and the development of layer pattern design methods for multi stream plate-fin heat exchangers. Zargoushi et al. [9] used the computational fluid dynamics (CFD) technique for investigating the impact of different fluid distributors on the rates of heat and mass transfer, in an industrial complex cold box equipped with plate-fins. Different side distributors were used, and their performance was evaluated by CFD. They found that using each of

these types of distributors led to different results in the heat transfer rate and the flow uniformity index of the cold box.

2. Problem description

The South Pars Gas Refinery in Phase 19 had a problem with its cold box, which showed an increase in pressure drop two years after its start. The increasing pressure drop threatened to damage the heat exchanger and caused numerous problems such as reduced NGL recovery yield, loss of revenue due to the decreased NGL production, and shutdown of the train for cleaning purposes. The use of joule-thomson throttling valves for refrigeration in the absence of turbo expanders in the ethane recovery unit caused temperature drops to -47°C, leading to ethane and propane carryover with the overhead methane product. The feed line pressure drop in the cold box was caused by several factors such as dryer bed malfunction, water entrainment, and deposit formation. Several attempts were made to solve the problem, including hygrometer tests and chemical washing, but to no avail. Finally, it was decided to perform chemical washing of the cold box using xylene solvent with flushing before and after washing.

Before cleaning a cold box heat exchanger, it's important to consider the level of fouling, the type of foulant, the compatibility of materials and components with cleaning chemicals, environmental regulations, accessibility for cleaning, and cost factors. Both mechanical and chemical methods can be used for deposit removal, such as manual cleaning, jet cleaning, and thermal cleaning, and so on. Mechanical cleaning has the advantage of being easy to use and can clean even completely blocked tubes, but it can also damage the equipment. Chemical cleaning uses chemicals to dissolve or remove deposits and has the advantage of providing uniform cleaning, being capable of cleaning inaccessible areas, and having moderate cleaning costs with longer intervals between cleanings. However, chemical cleaning must be done off-line and the unit must be isolated from the system.

Demerits of chemical cleaning are chemical used for cleaning are often hazardous to use and require elaborate disposal procedures; noxious gases can be emitted from the cleaning solution from unexpected reactions; chemical cleaning corrode the base metal and the possibility of excess corrosion cannot be ruled out; complete washing of the equipment is a must to eliminate corrosion due to residual chemicals.

Xylene solvents has high solubility for heavy hydrocarbons such as wax compounds and deposits [10]. Table 1 shows laboratory report about scales deposit analysis for two sampling points, collected from before and after the feed line strainer. From the deposit composition the mechanism for the formation may be derived. Analysis of the foulant (although possible) raises a number of issues. The first issue is related to the deposit recovery procedure which should be designed so that the recovered deposit is a representative sample of the material (the foulant) under investigation [10]. In addition, heat exchanger deposits are usually collected from the refinery after a long running time, which implies that a large number of different crudes would have traveled through pipelines, making it impossible to relate foulant composition with the properties of the feed material. In addition, the chemical and physical properties of heat exchanger deposits also change with temperature and aging. At higher temperatures, deposit structures become more carbonaceous with aging. The scale deposits description was wet odor deposit with black color.

After consulting the cold box vendor (Fives cryogenic) it was decided to plan chemical washing by xylene during 2018 refinery turnaround schedule. Aromatic solvents are recommended cleaning solvent for foulants consists of heavy organic deposits such as waxes and asphalts, etc. [5]. The purchased xylene solvent was mainly a mixture of orto-xylene, para-xylene and metha xylene with specification described in Table 2.

Table 1. Result analysis of sample scale deposits

	After Strainer	Before Strainer
Loss on drying (Moisture & Volatile matter) @ 110oC	45.4%	51.03 %
Test for H_2S	Positive	Positive
Analysis on dried sample:		
Loss on ignition (Organic Matter, Sulphur, etc.) @4500 oC	63.04 %	63.04 %
Ash content (Ignition @ 850oC)	24.56 %	31.54 %
Acid insoluble ash (SiO_2, etc.)	3.28 %	5.77 %
Iron Oxide as Fe_2O_3	17.47 %	24.67 %
Calcium Oxide as $CaCO_3$	0.18 %	0.09 %
Magnesium Oxide as $CaCO_3$	0.04 %	0.03 %
Sulphates as SO_4	Trace	Trace

Thanks to phase 19 neighbors' petrochemical plants xylene solvent are easily available at low cost and transportation. The novelty of the method proposed in this paper was in combining the back puffing and washing methods together and the initiative in using this method according to the strict design limitations and at the same time the time limitations to take the unit out of service .Furthermore, this approach offered additional benefits as it required no alterations to the existing design. By simply executing a single operation, conducting calculations, and preparing numerous instructions, the desired outcome was achieved at a remarkably affordable cost within a suitable timeframe. It is worth noting that Phase 19 stands out among the limited number of refineries that diligently implemented this method following thorough research, thereby reaping the advantages it had to offer.

Caution: This chemical washing method is recommended as a solution for gas refineries and processes that have the same limitations in design and operating conditions, such as the 10th Pars South Refinery, taking into account personal safety points and compliance with environmental issues. Of course, there are other solutions and each gas refinery has its own design structure, operating conditions and limitations, and all these points are influential in choosing a solution.

Table 2. Purchased xylene specification

Composition	Ethyl Benzene	Mehta Xylene	Benzene	Toluene	C9 Aromatics	Non-Aromatics	Total Sulphur	Total Chlorides	O & P Xylene
%	18	49	0.005	0.04	0.5	0.1	1 ppm (wt)	1 ppm (wt)	31.9

2.1. Safety considerations

The use of xylene solvents for cold box cleaning is hazardous and requires proper disposal to prevent environmental contamination and the emission of harmful gases from the cleaning solution. Safety procedures have been established for every step of the washing process. All personnel involved in the process must be trained in safety procedures, equipped with personal protective equipment (PPE), and aware of the hazards. Material safety data sheets (MSDS) should also be readily available in case of chemical exposure. Special attention must be paid during the steps of loading the solvent into the drum and evacuating the xylene, as these steps involve direct exposure and the possibility of leakage from the connection, pumping, evacuation, and disconnecting of hoses and fittings. Tight safety considerations were implemented. The washing path was a closed cycle and the procedures was batch circulation. After chemical washing, solvent draining was done carefully to the sump and was guided to off spec tanks.

3. Simulation procedure

According to available access points and the paths needs cleaning, A closed cycle was proposed for solvent back up and circulation, and its volume was measured first by methanol liquid fill up.

Cold box chemical washing procedure obeyed following step:

1- Unit shut down and depressurizing and draining of feed flash drum and all the connected lines;

After unit shutdown and accomplishment of cold box warm up, feed flash drum and all connected lines to it were drained and purged by nitrogen. For more safety and due to xylene high volatility, feed flash drum was completely purged and prepared to use as a xylene storage drum to avoid xylene vapors dispersion to environment and other consequent risks.

2- Feed line flushing;

After lines draining and depressurizing from hydrocarbons, the lower strainer of the feed lined was opened (strainer B in Fig. 3) and the opening of related path was enclosed by layers of plastics and flushing operation was done up to 3 barg pressure. It is expected to evacuate dusts that associated with gas stream by this way. Flushing operation was repeated three times at this step.

3- Xylene accumulation in feed flash drum;

After feed flash drum draining and purging, pneumatic pump and its related fitting and special 2-inch hoses was used to fill feed flash drum with xylene solvent. 30 m^3 xylene solvent was stored in the drum for each gas train.

4- Chemical washing of cold box by xylene;

According to the engineering staff calculations about 18m^3 xylene solvent per gas train was required. Fig. 3 illustrates schematic of configuration of xylene solvent charging to the drum and important fitting points. As xylene solvent injection to the drum was done via a temperature transmitter tapping point located on the feed line outlet path and exits through the 2 inches flange of feed line. The status of inlet and outlet location in the site such a way that the entering feed line is upper than exited feed line. Xylene solvent flows into the system as counter current compared to the gas flow in normal condition. The flow path has shown in Fig. 3 with arrows.

The equipment and fittings used to make xylene circulation loop are as follows:

a- Inlet manual valve, inlet strainer, pneumatic pump and gaskets;

b- Two 2-inch spools for the temperature transmitter connection and 2-inch path for xylene evacuation

c- An installed 2-inch valve at the top of the feed flash drum for pump isolation and evacuation of relating paths from xylene solvent.

d- An installed nipple spade on the drum inlet to establish xylene solvent loop. The nipple is required to xylene solvent evacuation between temperature transmitter point and the drum.

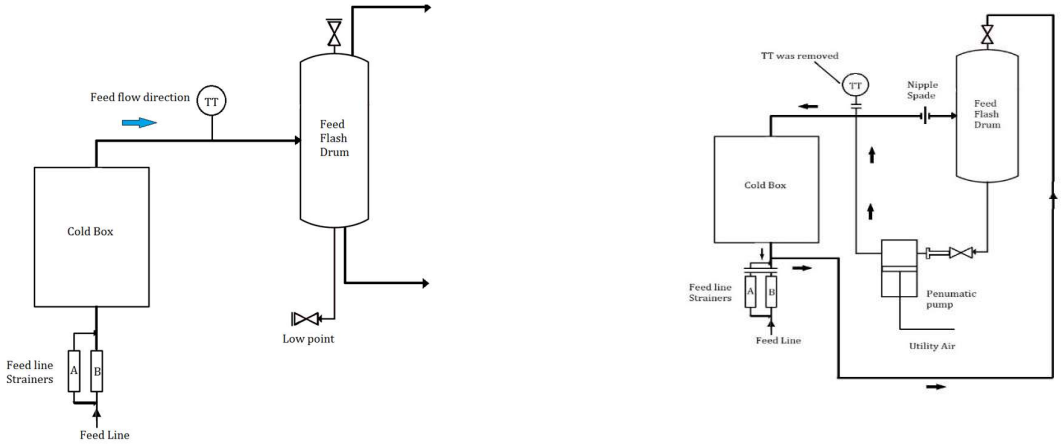
5- Xylene evacuation and lines drying;

When xylene solvent circulation has accomplished, it's time to evacuate it. At this step xylene solvent existed in the drum and paths was completely evacuated to the hydrocarbon sump where designed to collect all drained hydrocarbons in ethane recovery unit. Xylene solvent in the sump was pumped to off spec tank.

6- Repetition of feed line flushing.

After xylene solvent evacuation, all fittings and hoses were removed and disconnected, then Nitrogen was used for purging the loop. After ensure the loop was completely dry, flushing operation was repeated twice with at least 50

layer of plastic enclosed feed line opening. Feed line strainers was installed after loop evacuation, drying and cleaning. Finally, the unit was prepared for startup. After accomplishment of xylene circulation (washing process), solvent samples were taken and sent to the laboratory.



(a) (b)

Fig. 3. Schematic configuration of xylene solvent circulation loop: (a) Before; (b) after establishing xylene wash loop; arrows show xylene circulation path where flows as counter current rather than feed gas flow direction

4. Results and discussion

Results of cold box xylene wash for all gas trains will be discussed in this section. Subsequent graphs d/ata includes two parts: part one belongs to 45 days duration of normal operation before refinery turnaround from 14.07.2018 to 29.08.2018 and part two is for 1 month duration after refinery turnaround. Fig. 4 shows data timeline according to the refinery turnaround schedule

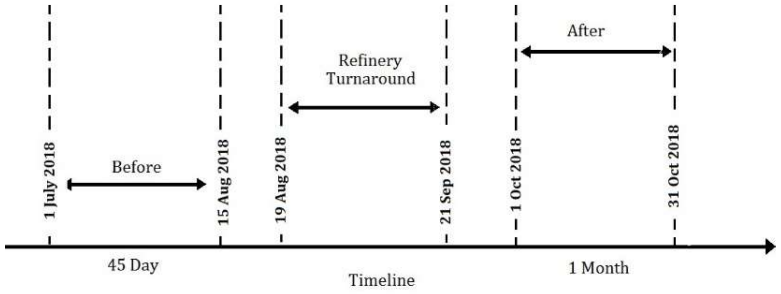


Fig. 4. Data collection timeline and phase 19 refinery turnaround schedule

The data comparison trends shown in Fig. 5(a), (b), (c), and (d) highlight the impact of cleaning procedures on the cold box pressure drop for gas trains 1, 2, 3, and 4, respectively. Fig. 5(a) shows that for gas train 1, the cold box pressure drop for a feed flowrate of 416 tons/hr on 14.07.2018 was 3.04 bar. After a refinery turnaround on 16.09.2018, the cold box pressure drop for a feed flowrate of 470 tons/hr was 2.64 bar and for 420 tons/hr was 2.16 bar. This indicates that at approximately the same feed flowrate, the pressure drop decreased by approximately 29%. Additionally, after a chemical washing of the cold box, it was possible to increase the feed flowrate to 480 tons/hr. These data comparisons highlight the importance of regular cleaning procedures in maintaining optimal plant performance and avoiding pressure drop. By cleaning the cold box, the plant can operate at higher feed flowrates and

reduce the risk of performance issues. These results demonstrate the significance of properly diagnosing the source of fouling and implementing effective cleaning methods to ensure optimal plant operation and efficiency.

In Fig. 5(b) same manner could be seen for gas train 2. At 17.08.2018 before xylene wash for 405 ton/hr feed flowrate, DP was 3.08 bar meanwhile after chemical washing DP was 1.81 bar for 402 ton/hr feed flowrate at 10.10.2018 which shows 41% decrease in cold box pressure drop.

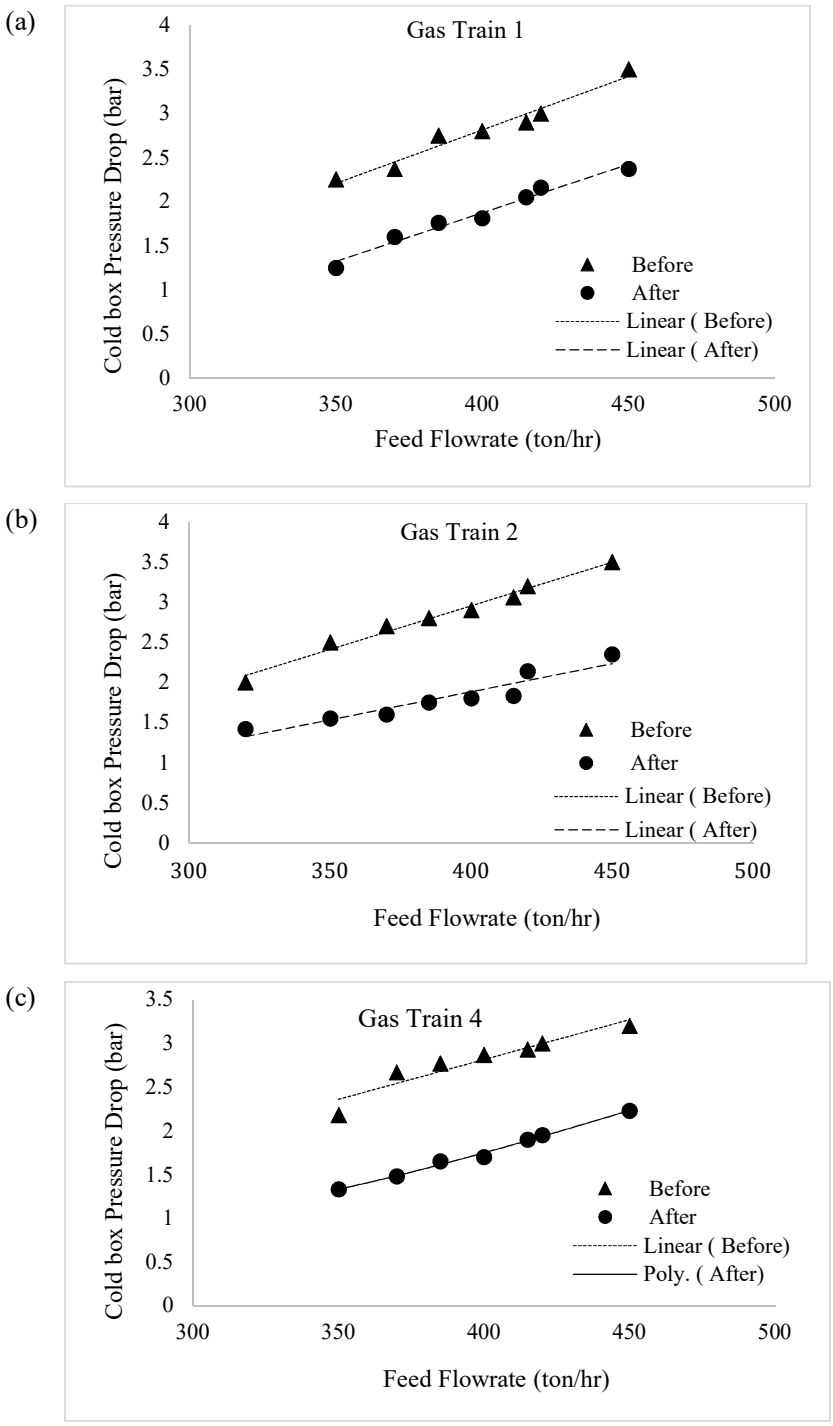


Fig. 5. Cold box DP (bar) versus feed flowrate for three different gas trains (a,b,c) before and after turnaround

As Fig. 5(c) demonstrated Gas train 3 experienced 33% improvement in DP. At 23.09.2018 after refinery turnaround for 445 ton/hr feed flowrate DP was 1.74 bar while the DP was 2.59 bar before xylene wash. Gas train 4 also has obeyed this manner of improvement. At 426ton/hr feed DP was 2.94 bar at 02.08.2018 and 1.97 bar at 20.10.2018, so 33% pressure drop was improved. Each gas train exposed to flushing operation at least for 5 times. Three times before chemical washing and twice after that.

As mentioned before 120 m^3 xylene solvent was predicted for chemical washing of cold boxes in all four gas trains. The cost of buying this amount of solvent was about 60,000 USD at that time. Other costs of xylene wash project was not financially as important as purchasing xylene for the refinery since it was owner of the used equipment, hoses, fittings and also it was a part of the plant turnaround schedule. In addition, used solvent was sent to Storage tanks unit for further treatment to reuse and storage. After chemical washing of cold box it was possible to increase each gas treating unit feed flow rate to 480 ton/hr in normal upstream conditions with acceptable cold box pressure drop lower than 3 bar. Totally for all four gas trains production rate could increase about 188ton/hr according to the Table 3. Assuming 0.2 USD/stm^3 price for sales gas, the amount of 188ton/hr or 5.3$mstdm^3$/day provides 387 million USD/year revenue for the refinery which is impressive in comparison with the project costs.

Table 3. Increases in gas trains production rate

GTU	Max. feed flowrate before washing	Max. DP (bar) before washing	Feed flowrate till 480 ton/hr
GTU 1	415 (480)	3.01	65
GTU 2	405 (480)	3.08	75
GTU 3	480 (480)	2.95	0
GTU 4	432 (480)	3.02	50
total	1732 (1920)	-	188

5. Conclusion

The 10th refinery of the South Pars Gas Complex in Iran had a notable decrease in production rate because of deposit formation in the gas treating unit multi stream heat exchanger (cold box). Diagnosing the source of fouling in a cold box heat exchanger is key to preventing pressure drop. An appropriate cleaning method was selected after comprehensive study and teamwork of engineering, production, laboratory and HSE experts. Flushing and xylene wash are two recommended cleaning methods to avoid pressure drop and debottlenecking issues during shutdown. These methods can increase gas flowrates and provide economic benefits. Proper cleaning procedure provided for xylene wash to remove deposits and maintain optimal plant operation while avoiding costly shutdowns. Xylene wash is effective, safe, and cost-effective, as it can be performed off-line without the need to dismantle the unit. 10th refinery of the south pars known as SPGC phase 19 experienced favorable results after xylene wash of cold box about 188 ton/hr increase in total production rate and the purpose of conducting this study was sharing considerations and details of the method as successful industrial experience for similar plants. In conclusion, diagnosing the source of fouling and implementing effective cleaning procedures is crucial for maintaining the performance and efficiency of a cold box heat exchanger. The use of flushing and xylene wash is an effective solution for preventing pressure drop and achieving economic benefits for plant operation. By combining these tools, plants can ensure optimal performance and avoid costly shutdowns or performance issues.

Nomenclature

Barg	Bar gauge
DP	Differential Pressure

GTU	Gas Treating Unit
CFD	Computational Fluid Dynamic
NGL	Natural Gas Liquids
MSDS	Material Safety Data Sheet
SPGC	South Pars Gas Complex

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