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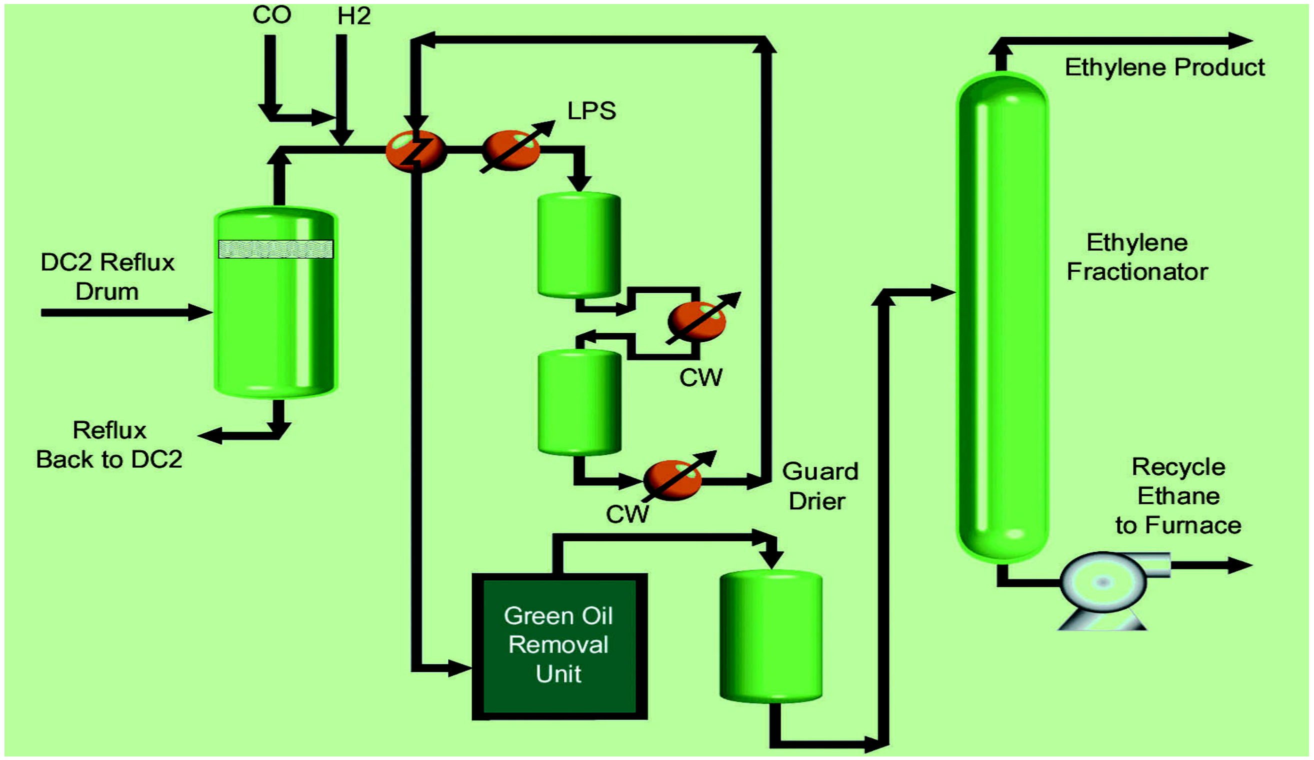

Back End C2 Hydrogenation for Ethylene Production

REPRINT

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A comparison of separation methods for Green Oil in ethylene production

Green oil is an oligomer formed in all C2, C3 and C4 hydrogenation reactors of ethylene plants and other petrochemical production facilities. Green oil is a mixture of C4 to C20 unsaturated and reactive components with about 90% aliphatic dienes and 10% olefins plus paraffins. In the C2 acetylene hydrogenation reactor where acetylene is hydrogenated to ethylene and ethane, the most commonly used catalyst is palladium (Pd) on Alumina (Al_2O_3) support. Green oil polymer is formed by side reactions of the hydrogenation reaction itself, and it cannot be totally avoided. The polymer formation starts by dimerization of acetylene with hydrogen to butadiene followed by oligomerization with successive addition of acetylene molecules to a base chain molecule adsorbed to the Pd surface.

The low molecular weight fraction of the green oil vaporizes into the gas stream, while part of the heavier fraction deposits in the pores of the catalyst. The rest of the heavier fraction is carried away with the gas as fine droplets mostly < 5 micron size with concentrations of green oil in the gas in the order of 100 ppmv to 1,000 ppmv depending on the operating temperature, age of the catalyst, CO content, H_2/acetylene ratio, etc.

The gas leaving the hydrogenation reactor is cooled, and more green oil condenses into fine droplets, which deposit on the downstream heat exchangers, dehydrator beds, and on the ethylene fractionator internals. These depositing droplets are polymeric and cause fouling of the equipment, thus potentially leading to expensive unplanned shutdowns to clean up the deposited green oil.

Fuel gas used for the regeneration of the dehydrators strips out the deposited green oil on the molecular sieves; the fuel gas thus becomes contaminated with the green oil. This contaminated fuel gas then potentially causes fouling of the furnace's low NOx burner nozzles leading to lower furnace efficiency and more frequent and costly burner tip cleaning.

Different industrially used methods for the separation of green oil from the hydrogenation reactor gaseous effluent stream were evaluated including:

- Washing of the wet gas stream from the reactor with a liquid ethylene stream in a stripping tower,
- Impaction of the wet gas through a packed bed,
- Separation by a mesh pad in a knock-out drum,
- Use of a high efficiency liquid-gas coalescer with specially formulated and designed filter media - the Pall liquid / gas coalescer.

Of the separation options investigated, the Pall high efficiency liquid / gas coalescer was found to be the most cost effective option that achieves a suitable and optimized degree of green oil removal from the ethylene-ethane stream.

Introduction

In petrochemical steam crackers, acetylenes (acetylene, methyl acetylene) are contaminants in the ethylene and propylene products. These acetylenes cannot be separated from the ethylene and propylene products by fractionation because of their close volatility to ethylene and propylene; thus, acetylenes are typically removed by selective hydrogenation to olefins or non-selective hydrogenation to paraffins.

Focusing on removing acetylene to meet the typical ethylene specification of less than 1 ppmv, the acetylene hydrogenation reactor is placed either within the cracked gas (CG) compression train (front end hydrogenation) or downstream between the back end deethanizer and the ethylene fractionator (back end hydrogenation). The most used catalyst for acetylene hydrogenation is Pd on Al_2O_3, which is selective to hydrogenation of acetylene to ethylene and not to ethane, even at a high partial pressure of H_2.

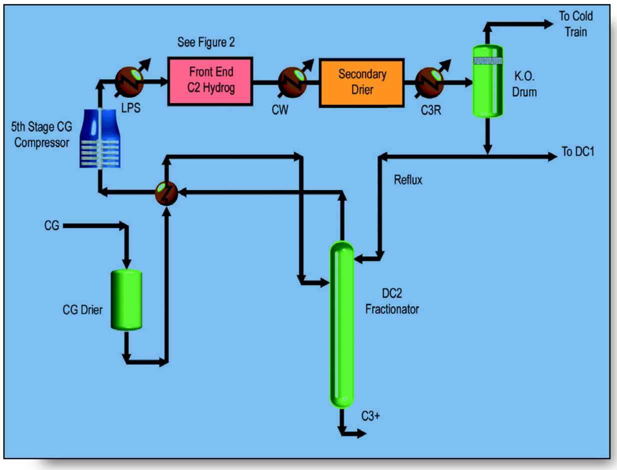

Fig 1. Front End Hydrogenation Unit

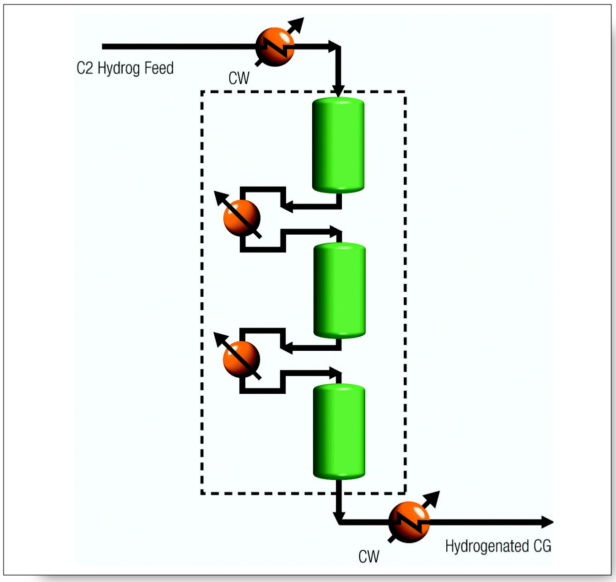

Fig 2. C2 Hydrogenation Reactors

Front End Hydrogenation

In the ethylene compression train and downstream of the cracked gas (CG) drier, a front end deethanizer (DC2) is used in plants cracking gaseous feedstocks, or a front end depropanizer (DC3) is used in plants cracking liquid feedstocks. The overhead of the DC2 or the DC3 containing the light components of the CG is sent to the gas phase acetylene hydrogenation unit (C2 Hydrog), as shown in figure (1) and figure (2).

Back End Hydrogenation

The acetylene hydrogenation unit treats the overhead of the back end DC2, which contains ethane, ethylene, and some 0.5-2.5% acetylene. In this configuration, H_2 has to be added, as all the H_2 present in the CG would have been removed in the chilling train and the demethanizer (DC1) upstream of the reflux drum (DC2), as presented in figure (3).

Green Oil formation

Green oil polymer is formed by side reactions of the hydrogenation of acetylene to ethylene and ethane over the Pd catalyst. It occurs due to the dimerization of acetylene to butadiene followed by oligomerization with successive addition of acetylene to a chain of molecules adsorbed on the Pd surface. The green oil is a mixture mainly comprised of C4-

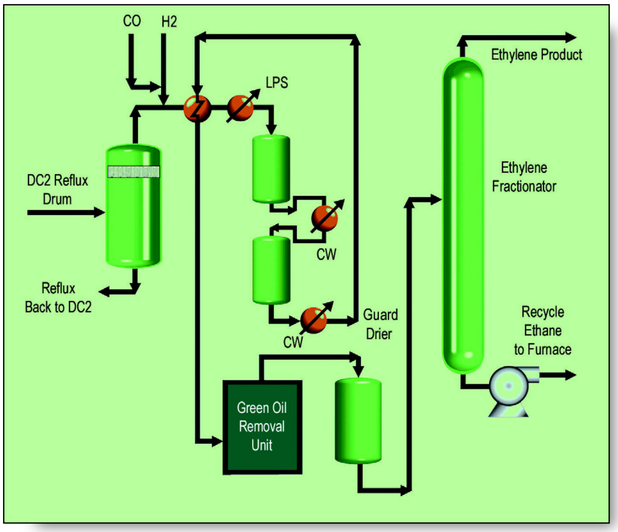

Fig 3. Back End C2 Hydrogenation

C20 reactive oligomers of varying composition, with boiling point (B.P.) range from 120 to 400°C. The heavier fraction stays adsorbed on the catalyst pores causing eventual loss of the catalyst activity and thus requires regeneration by steaming out the deposited green oil. The light end components of the green oil remain in the gas phase, part of which condenses into fine droplets in the gas stream as it cools leaving the reactor. These fine droplets cause fouling of the downstream equipment.

Green oil formation is decreased by the use of silver promoted Pd catalyst on Al_2O_3, which helps terminate the chain growth at the butadiene stage. Thus, instead of the formation of heavier diolefin oligomers, the light butadiene simply exits with the gas. This new catalyst generally reduces the formation of green oil to a third or half the amount formed with the non promoted catalysts. The concentration of the green oil in the gas leaving the hydrogenation reactor is in the order of 100 ppm-1000 ppm depending on the operating temperature, age of the catalyst, CO content of the gas, H_2/acetylene ratio, etc. The droplet size of the green oil condensing in the gas stream downstream of the reactor is mostly less than 5 micron in size. Aerosols formed from the condensation mechanism are known to have small drop size distributions in the range of a few microns[1].

The amount of green oil formed is primarily a function of the concentration of acetylene being converted and the temperature resulting from that conversion. Hence, the rate of formation of green oil is higher in the lead bed. Green oil formation decreases with increased partial pressure of H_2, which is the main reason that much less green oil is formed in front end hydrogenation units compared to back end hydrogenation units. Typically, in back end hydrogenation reactors, 10-20% of the acetylene is converted to C4 and heavier green oil.

Process related problems caused by Green Oil

Ethylene plants are expected to operate for a period of 5-7 years between turnarounds. Hence, a spare bed is provided to allow the plant to continue operating when one of the beds is fouled. The fouled bed is taken out of operation, and the spare bed is put into operation. The green oil is then drained and the fouled bed is regenerated and put on standby mode.

The green oil droplets carried out with the hydrogenated gas from the reactor deposit and cause fouling of the downstream heat exchangers, dehydrator beds, and ethylene fractionator internals, thus eventually requiring expensive unplanned shutdowns to clean up the deposited green oil.

Also, fuel gas used for the regeneration of the dehydrators, strips out the deposited green oil on the molecular sieves, contaminating the fuel gas with the green oil. The contaminated gas causes fouling of the furnace's low NOx burner nozzles, which leads to lower furnace efficiency and more frequent and costly burner tip cleaning.

Removal of Green Oil from gases

Many methods have been adopted to remove green oil from the hydrogenated ethylene/ethane stream including:

Wash Tower: The wet gas stream from the reactor is contacted and washed counter - currently with a liquid ethylene stream from the ethylene fractionator in the green oil wash tower (Figure 4). The contaminated ethylene liquid from the bottom of the wash tower is then recycled back to the DC2 tower. This method is effective in removing the green oil, but expensive as it adds a recycle stream that is typically 5 % of the net stream, thus reducing the capacity of the fractionation system.

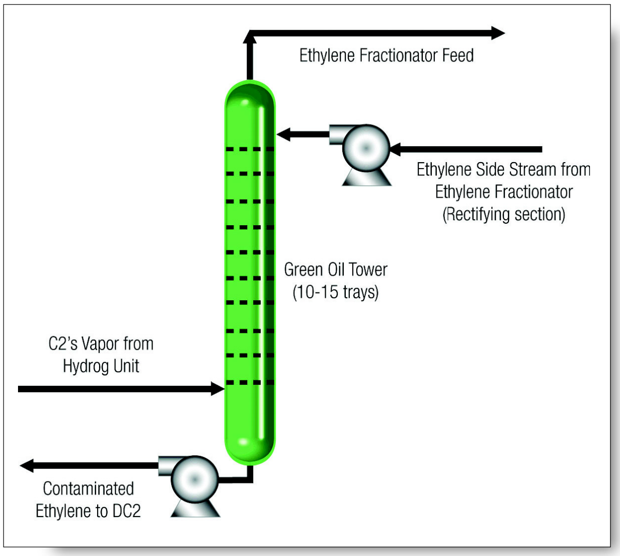

Fig 4. Green Oil Tower Unit

Packed Bed: The green oil droplets in the gas stream impact against the packing (pellets or beads) surface because of their inertia. After a number of droplets are caught on the packing surface, they agglomerate and tend to drain along the packing surface. The collection efficiency is a function of superficial bed velocity, droplet size, bed packing particle size, the tortuosity of the gas flow path through the bed and other system conditions such as the liquid and vapor properties. The number of turns or tortuosity of gas stream path through the bed is a function of the bed length and the packing size.

Analysis of the packed bed[2] performance shows increasing droplet removal efficiency when the packing size is decreased, but this is at the expense of an increased pressure drop. The efficiency can also be improved by increasing the gas velocity through the bed up until a maximum threshold. Well designed beds can achieve 80-90% green oil removal, and are rated at 99% removal of drops > 1 micron size. However, the packed bed separator (Figure 5) is sensitive to reductions in the flow rate to below design conditions (increased turn down ratio) as the mechanism of separation is inertial impaction, which is a function of gas velocity. Therefore, at reduced flow rates, the efficiency of the packed bed separator will be adversely affected.

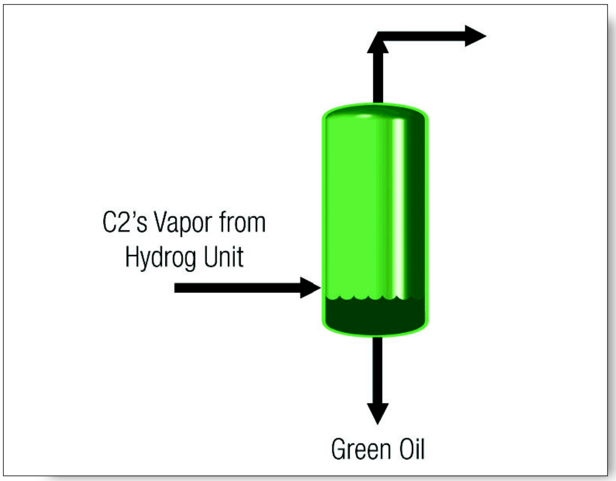

Fig 5. Packed Bed Treating Unit

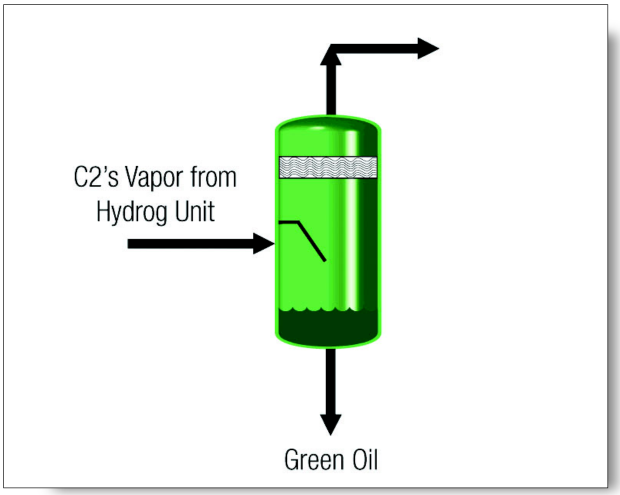

Fig 6. KO Drum with Mesh Pad Unit

Mesh Pad: A coarse bed of fiber material that is configured in a vertical flow knock out vessel is used and has a reasonable removal efficiency of 95 % for drops > 5μm. For smaller drops, the removal efficiency is greatly reduced. The overall green oil separation is in the range of 70 – 80 %. The mesh pad (Figure 6) also operates on the mechanism of inertial impaction to separate out the aerosol drops. Therefore it is also subject to the limitations of reduced flow rates to below design conditions and cannot tolerate a high turn down ratio without significant loss of performance.

High Efficiency Liquid / Gas (L/G) Coalescer: Specially formulated and designed filter media coalescer, as demonstrated by the Pall L/G Coalescer (Figure 7), is used to capture very small aerosols and combine them into large, more easily separated drops. High efficiency L/G coalescers have achieved 98-99% total green oil removal and are rated at 99.98% removal of drops > 0.3 μm[3-5]. Here, the removal mechanism is based on diffusion or Brownian motion of the aerosol drops in the coalescer media. This mechanism allows for excellent separation even at lower than design flow rates, therefore the high efficiency liquid / gas coalescers can overcome high turn down ratios.

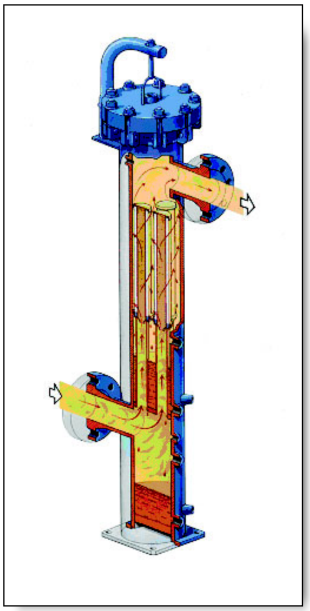

Figure 7. Pall SepraSol™ High Efficiency Liquid / Gas Coalescer

Comparison of the Green Oil Removal Units

For an ethylene-ethane vapor stream of 100 tons/hr at a pressure of 20 kg/cm^{2}a, the following table compares the units used for green oil removal in acetylene hydrogenation systems.

	Units	Wash Tower*	Packed Bed*	KO Drum*	Pall Coalescer
Vessel Diameter	mm	2000	2000	1700	700
Vessel Length	mm	9000	3500	2500	3500
Number of Trays		10	NA	NA	4 SepraSol™ Plus Coalescers
Packed Height	mm	NA	2000	NA	NA
Wash Ethylene Rate	Ton/h	5	NA	NA	NA
Δ P Across Unit	kg/cm^2	0.1	0.5	0.01	0.2
Green Oil Droplet Removal	%	100%	99% of drops > 1.0 µm	95% of drops > 5.0 µm	99.98% of drops > 0.3 µm
Overall Green Oil Removal Range	%	99-100	80-90	70-80	98-99

** Based on experience of Stone & Webster*

From this comparison we see that the wash tower provides the highest degree of green oil separation removing 100 % of any sized drops and will also remove some green oil in the vapor form. However, it also has a high operating cost (5 % ethylene recycle stream) and requires the largest capital investment for the tower. Therefore, the best separation option was found to be the high efficiency liquid / gas coalescer system as it had a removal efficiency of 99.98 % for aerosols > 0.3 µm and had a modest capital investment with low operating costs. This separation option also maintains its high level of removal efficiency even at flow rates lower than design because the coalescer's capture mechanism is based on diffusion. Lower overall green oil separations of 98-99% can be explained by the presence of some green oil in the vapor state that the coalescer is not designed to remove. A more in depth discussion of the technology offered by Pall Corporation for high efficiency liquid / gas coalescers is given below.

High Efficiency Liquid / Gas Coalescer Technology

The separation of liquid aerosol contamination with high performance liquid/gas coalescer cartridge systems has found widespread acceptance in refinery and gas plants in recent years for a number of applications$^{6-8}$, including protection of compressors, turbo equipment, burner nozzles, amine and glycol contactors, molecular sieve beds, and hydrotreater catalyst beds. This has largely been the result of traditional separation approaches including knock out vessels, centrifugal separators, mesh pads or vane separators not meeting the end user's requirements for aerosol reduction. The primary rational for the use of high efficiency coalescers is that aerosol contaminants are in the sub micron and low micron size range^3.

Another benefit of the liquid/gas coalescer is that this type of separation device can be operated at significantly lower flow rates than the initial design flow rate, which means that it has a high turn down ratio. This is due to the fact that the separation mechanisms are based primarily on diffusion and direct interception unlike vane separators and mesh pads that rely heavily on inertial separation principles. This allows the high efficiency liquid/gas coalescer systems a greater degree of flexibility, and they can operate at peak performance even for reduced flow rates that can occur during commonly encountered partial plant shutdowns and upset conditions. Generally, the high efficiency liquid/gas coalescers are used for inlet aerosol concentrations of less than 1,000 ppmw (0.1 %) and are placed downstream of other bulk removal separators as the final stage. Outlet concentrations for these high efficiency liquid / gas coalescers are as low as 0.003 ppmw$^{3-5}$.

High efficiency liquid/gas coalescers are generally constructed from glass fibers since this material allows for a fine porous structure with fiber diameters of a few microns. The small pore size is needed to achieve greater capture and separation of these fine aerosols. The use of a innovative surface treatment[9] on high performance vertical liquid / gas coalescer cartridge systems has been proven to enhance performance significantly by allowing higher flow rates or smaller housing diameters compared to untreated coalescers.

A Pall vertical high efficiency liquid / gas coalescer system is depicted in Figure 7. The inlet gas with liquid aerosol contamination first enters at the bottom of the housing into a first stage knock out section. Here any slugs or larger size droplets (approximately > 300 μm) are removed by gravitational settling. The gas then travels upward through a tube sheet and flows radially from the inside of the cartridges through the coalescer medium to the annulus. The inlet aerosol distribution is in the size range of 0.1 μm – 300 μm, and after passing through the coalescer medium, it is transformed to enlarged coalesced droplets in the size range of 0.5 - 2.2 mm.

The advantage of flowing from the inside to outside of the coalescer cartridge is that the gas velocity can be more easily adjusted in the annulus by selecting the optimum housing diameter to prevent re-entrainment of coalesced droplets. As the gas leaves the coalescer cartridge and travels upward in the annulus it contributes to the total flow, thereby increasing the annular velocity. The annular velocity is modeled as a linear function with vertical distance; the annular velocity is zero at the bottom of the cartridge and increases to a maximum value at the top of the cartridge.

Four steps have been identified with the mechanism of the formation and removal of droplets in the coalescer medium:
1) Capture
2) Coalescing
3) Release
4) Drainage

The formation of the large coalesced drops first involves the capture of the small aerosol droplets onto the fibers of the coalescer medium. The capture of the small aerosol droplets is due to diffusion or Brownian motion that causes the droplets to have a random motion that leads to impacting the fibers in the coalescer media. The actual coalescing or merging of the fine droplets takes place on the fibers and especially at fiber intersections. The coalesced droplets are then released from the fiber due to the drag force of the gas flow exceeding the adsorption energy. This process is repeated through the depth of the coalescer medium until the coalescing process is completed and the largest possible stable droplet size is achieved.

During the coalescing stages, the growing droplets are also draining downward inside the media pack due to the force of gravity. The application of the novel surface treatment allows the release and drainage process to proceed at a faster rate, which in turn frees up more coalescing sites on the fibers and allows the coalescer to process higher inlet liquid aerosol concentrations than an untreated coalescer medium. The surface treatment greatly enhances this drainage, and as a direct consequence of the treatment, the coalesced droplets are shielded from the upward gas flow in the annulus for most of the length of the coalescer cartridge.

The coalesced droplets are first exposed to the annular gas flow when they appear on the external face of the coalescer medium pack at the bottom third of the coalescer cartridge (See Figure 8a). Once the coalesced droplets are released to the annular space they are subject to the force of the upward flowing gas. The trajectory of the coalesced droplets is modeled on a force balance between gravity settling and the drag force created by the gas flow past the droplets. This analysis leads to the calculation of a critical annular velocity for re-entrainment.

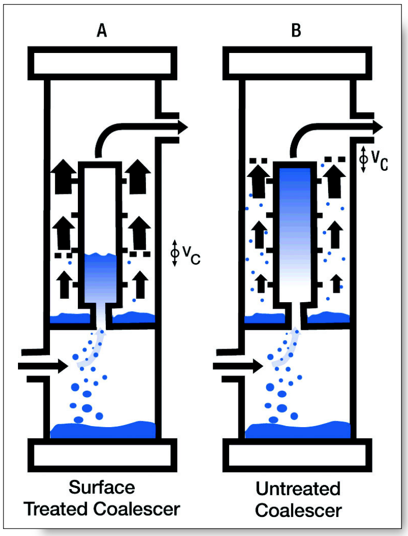

Figure 8a and 8b. Effect of Surface Treatment on Annular Velocity

Due to the surface treatment, there are minimal coalesced droplets present in the annulus above the drainage point at the bottom third of the coalescer cartridge. For a coalescer cartridge that is not specially surface treated, the coalesced liquids are present throughout the length of the coalescer in the annulus space, and the critical annular velocity for re-entrainment is given for the top of the element (See Figure 8b). For the treated coalescer, it is allowable to have annular velocities greater than the critical value for re-entrainment in the portion of the annulus space where there are no liquids present. This allows the maximum annular velocity at the top of the coalescer cartridge to be about three times the critical re-entrainment value needed at the vertical position of the lower one third of the cartridge height where liquids are present.

Therefore, the maximum annular velocity at the top of the coalescer cartridge is found to be about three times greater than the value for an untreated coalescer. The annulus area is determined using the maximum allowable annular velocity and designed to be of sufficient size to prevent re-entrainment and as small as possible to minimize the housing diameter.

Liquid/Gas Coalescer Construction - Surface Treatment

The liquid/gas coalescer is constructed of an inner rigid stainless steel core around which is placed the active pleated glass fiber coalescer medium. Using layers of increasing pore size tapers the pore structure in the coalescer medium. The inlet gas first encounters the smallest pores followed by increasing pore size with penetration distance to allow for more space as the coalesced droplets grow. The pleated coalescer medium is supported by a mesh structure to provide mechanical strength, followed by a coarse outer wrap that serves as a drainage zone. The entire coalescer cartridge is treated with an aqueous fluorocarbon emulsion that penetrates through the depth of the glass fiber coalescer medium and drainage layers leaving a thin fluorocarbon coating on all of the surfaces. The result is that the surface energy of the coalescer medium is lowered sufficiently to prevent most liquids from wetting out the coalescer fibers.

This treatment effectively creates a coalescer medium that is both hydrophobic (water repellent) and oleophobic (oil repellent). This effect can be characterized through use of contact angle measurements. In Figure 9, a droplet is placed on a surface treated glass fiber and an untreated glass fiber. The degree to which the droplet is spread out, or the fiber wetted, is measured by the contact angle of the liquid with the solid. For drops that are not strongly adsorbed to the solid surface, the contact angle is greater than 90 degrees while the untreated wetted surface has a contact angle approaching zero degrees. Another way to demonstrate this effect is to dip a section of the coalescer medium into a test liquid and compare it to an untreated coalescer section. The treated coalescer medium quickly sheds the liquid, while the untreated coalescer medium absorbs the liquid and acts as a sponge.

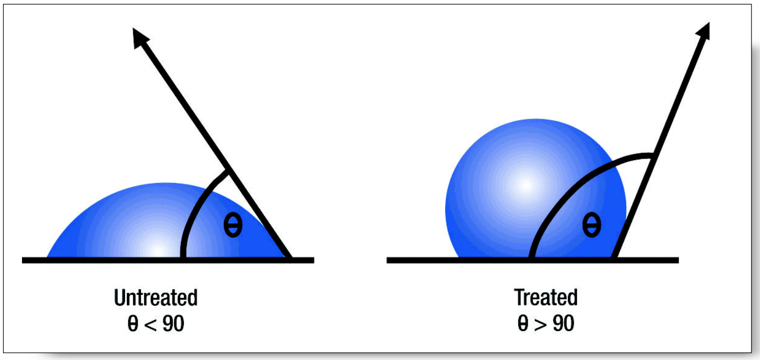

Figure 9. Contact Angle of Treated and Untreated Coalescer Medium

The amount that the liquid aerosols wet out the coalescer fibers has remarkable effects on coalescer performance. One such effect is capillary flooding which is illustrated in Figure 10. Liquid aerosols entering an idealized cylindrical pore made from untreated coalescer medium result in the liquids forming a continuous layer along the walls of the capillary. As more liquids enter the pore, the liquids coating the pore walls build up and eventually block the pore completely. The gas pressure then rises in the pore and ultimately causes the drop to be ejected from the pore in such a manner that the drop is atomized into a number of smaller droplets. These droplets are smaller than the largest drop size possible by coalescence, and are re-entrained by the annular flow. A surface treated coalescer pore behaves quite differently, and the liquids do not wet the capillary walls due to the weak interaction between the liquid aerosols and the surface treated

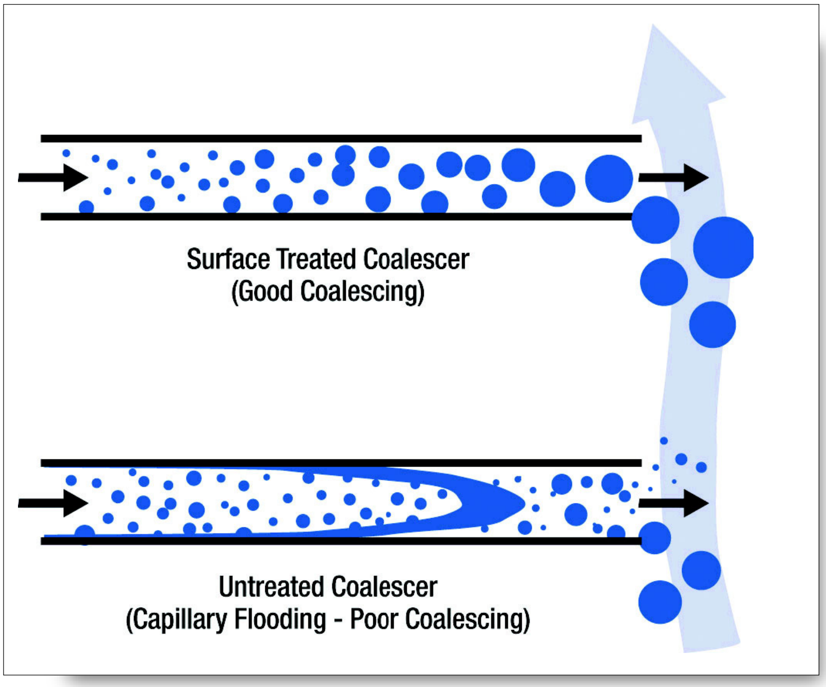

Figure 10. Effect of Surface Treatment on Media Velocity

pore walls. The drops instead tend to coalesce with each other throughout the length of the pore, and when they leave the coalescer medium are at the largest possible size by coalescence. The large drops then settle by gravity and are not re-entrained. It should be noted that, in the case of the treated coalescer pore, the walls of the pore do not become wetted out, and the capillary cross section is never blocked so that atomization does not occur.

Another effect of the surface treatment is that it provides the coalescer with anti-fouling abilities. Most of the solids in the gas are associated with the liquid aerosol droplets. The ability of the surface treated coalescer to repel these droplets and not wet out also prevents solid contaminants from adhering to the coalescer fibers. This allows the coalescer to provide an extended service life over non treated coalescers. A typical field service life encountered for the surface treated coalescer is from 12-24 months while traditional coalescers without surface treatment have been found, in specific cases, to last from 2 – 6 months.

The surface treatment also allows the coalescer to operate with less hold up volume of liquids, as they tend to drain quickly due to the low attraction between the coalescer fibers and the liquid drops formed. The result is that a less obstructed pathway is created for the gas passing through the coalescer, and consequently a lower overall pressure drop is experienced as compared to untreated coalescers.

The primary effect of the surface treatment is to enhance drainage of the coalesced liquids. This results in improved capability to handle higher inlet liquid concentrations, higher annular velocities and a lower pressure drop. High Efficiency Liquid / Gas Coalescers also operate on the separation mechanism of diffusion that allows them to tolerate high turn down ratios (greatly reduced flow rates from design conditions).

Conclusions

Several different technologies were evaluated for green oil removal from the hydrogenated cracked gas in ethylene plants including: ethylene wash tower, packed bed, knock-out drum with mesh pad, and high efficiency liquid / gas coalescer. Based on this comparison, the wash tower provided the highest degree of green oil separation with 99-100 % overall green oil removal, but also at the highest operation cost (ethylene wash steam rate is about 5% of the ethylene product rate) and required the largest capital investment for the tower. The packed bed required a lower capital investment and operating cost, but provided a lower separation quality with an overall green oil removal of 80-90 %. The knock out drum with mesh pad also offered a lower cost solution, but this option provided the poorest separation of only 70-80% overall green oil removal. The best separation option based on low operating cost and the smallest sized vessel, was the high efficiency liquid / gas coalescer system as it had a removal efficiency of 99.98 % for aerosols > 0.3 μm and an overall green oil removal of 98-99 %. High efficiency liquid / gas coalescers also have the advantage over other technologies of being able to tolerate high turn down ratios due to their diffusion based separation mechanism.

An in depth discussion of the technology offered by Pall Corporation for high efficiency liquid/ gas coalescers was presented. Advances in coalescer technology were discussed including details of the coalescing mechanism and how a chemical surface treatment can be used to enhance the coalescer separation ability.

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