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Novel Adsorption Distillation Hybrid Scheme for Propane/Propylene Separation

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Abstract

A novel adsorption–distillation hybrid scheme is proposed for propane/propylene separation. The suggested scheme has potential for saving up to ~50% energy and ~15–30% in capital costs as compared with current technology. The key concept of the proposed scheme is to separate olefins from alkanes by adsorption and then separate individual olefins and alkanes by simple distillation, thereby eliminating energy intensive and expensive olefin-alkane distillation. A conceptual flow schematic for the proposed hybrid scheme and potential savings are outlined.

INTRODUCTION

Propylene is produced in larger quantities than any other organic chemical in the United States except ethylene (*1*). Its main use is in the production of plastics. Propylene is produced as a by-product isolated from C_3-rich petrochemical streams. Currently, separation of the mixture is carried out by distillation which is very expensive and energy intensive because of the low relative volatility of these two compounds. Columns are typically up to 300 feet tall and can contain over 200 trays. The reflux ratios are greater than 10 and require a high energy input (*2*). Olefin purifications (including propane/propylene) are the largest consumers of energy for distillation in the chemical industry, using between 25 and 50 trillion Btu per year (*3*).

Propylene is generally produced as a coproduct in one of at least four different processes:

Steam cracking, in which propylene is a major coproduct to ethylene
Refinery gas streams primarily from three processes: fluid catalytic cracking (FCC), visbreaking/thermal cracking, and coking

Propane dehydrogenation (Houdry, UOP, etc.)
Olefin metathesis (Lyondell)

Worldwide steam cracking and refinery gas streams are the major traditional sources of propylene. Catalytic dehydrogenation of propane and olefin metathesis constitutes a minor, novel source of olefin. While metathesis technology is not being practiced due to unfavorable economics, catalytic dehydrogenation of propane seems to have gained acceptance in recent years. A propane dehydrogenation plant in Thailand started operating in 1990, and there are two new plants (one in Belgium and one in Mexico) under construction. The economics of catalytic dehydrogenation of propane is comparable with that of conventional sources, and depending on local conditions, may be favored for new propylene capacity. However, no propylene capacity based on propane dehydrogenation exists or is currently planned in the United States (*4*). Thus, with the exception of the Lyondell metathesis facility, all United States propylene capacity is associated with olefins plants (63%) and refineries (37%) (*5*).

With the exception of olefin metathesis technology, where no propane is used, all other methods of propylene production involve separation of C_3-rich streams followed by fractionation of a propylene/propane ($C_3^=/C_3$)

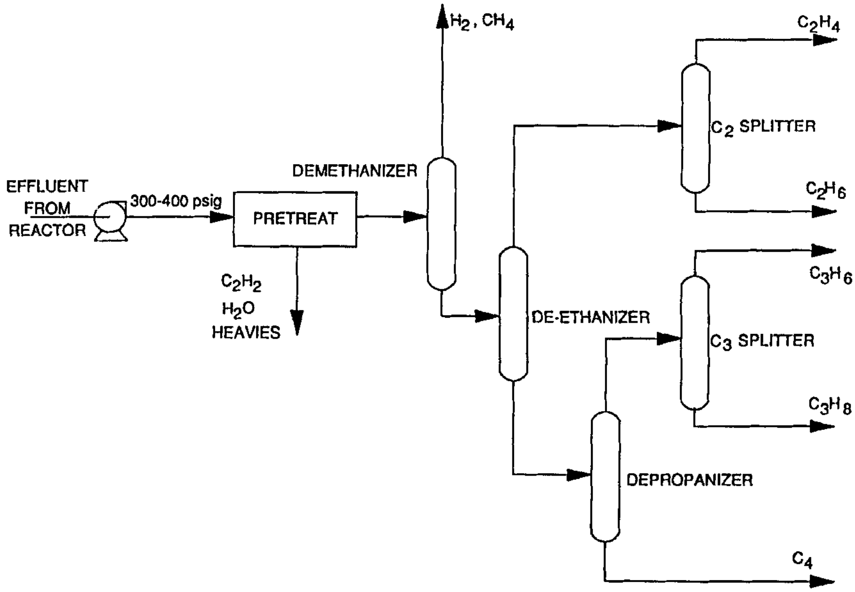

FIG. 1. Generic olefin separation plant.

mixture. Although there are differences in product composition between the gas mixture coming from steam crackers and that of refinery gases, the two streams are similar enough to allow processing according to the same general scheme and often in the same equipment. Figure 1 shows the plan of a generic olefin separation plant. The separation scheme for propylene produced via catalytic dehydrogenation of propane is somewhat simpler due to a less complicated product composition but still involves isolation of a C_3-rich stream followed by the fractionation of a C_3/C_3 mixture.

In all cases, propylene is obtained by the isolation of C_3-rich streams followed by rectification of the C_3/C_3 fraction.

The energy expenditure for the propylene separation is, therefore, a combination of two factors: 1) isolation of a C_3-rich stream, involving considerable compression, which is energy intensive; and 2) fractionation of the C_3 mixture. Fractionation of C_3s is difficult, energy intensive, and expensive because of the small difference in volatility between propylene and propane. An alternative, energy efficient method for effecting propylene separation would, therefore, be highly desirable. It is also apparent that a more efficient option to the entire olefin separation scheme (currently based on distillation technology) would be attractive.

LITERATURE REVIEW

There have been a number of prior attempts to address the problem of energy and cost savings in propane/propylene separation. Considerable effort was devoted to improve the distillation approach by using extractive distillation (*6*) or heat pump (*7*) concepts. These have met with limited success.

A number of more exotic approaches were proposed. A Podbielniak-type centrifugal separator fitted with a spiral rotor was reported to produce polymerization-grade propylene from propane/propylene mixtures at 1.1–1.5 kg/cm^2 (*8*). Separation by absorption in solutions containing silver (*9*) or palladium (*10*) cations was also proposed.

A novel approach has been proposed recently involving the use of a membrane hybrid system to affect the separation (*2*). However, materials for this approach are currently not available.

A more conventional, yet novel, approach to the problem is to use an adsorption distillation hybrid scheme as an alternative separation process. In fact, the adsorption approach to olefin and paraffin separation has a well-established precedence in the separation of higher hydrocarbons. There is considerable literature devoted to the problem, which includes commercial processes based on adsorption. For example, Universal Oil Products (UOP) has developed a process for selective adsorption of 1-butene from a feed mixture comprising 1-butene and other mono-olefins.

The process employs either Type X or Y molecular sieves. Union Carbide's OlefinSiv process uses type 5A molecular sieves primarily for the separation of *n*-butylenes from isobutylene. In this case the separation is based on a molecular sieve as in the case of *n*-paraffin separations (*11*). The OlefinSiv and the UOP processes operate in the gas phase, but UOP's Olex process separates olefins from paraffins in the liquid phase. The process was initially developed for separation of higher normal olefins ($>C_{10}$) from the corresponding *n*-paraffins. The separation is performed using modified X or Y zeolites. The technology was subsequently extended to the separation of propylene from C_3 mixtures (*12*). It was claimed that "the process can produce polymer-grade propylene at excellent extraction efficiencies," but no commercial applications of the technology followed. Part of the problem is that the process uses refinery-grade propylene as a feed, i.e., it is placed too far down the olefin separation scheme to offer real economic incentive for its deployment.

Currently, there is no commercial process for vapor-phase adsorptive separation of light olefins (C_2 or C_3). However, a number of adsorptive materials have been investigated toward this end. All the examples are based on modified molecular sieves; materials used for higher olefin separations.

The common pitfall of most of the studies is that they consider the adsorbent materials without an adequate process development effort to maximize adsorbent properties. It is very important to match the properties of the adsorbent to the process.

SUGGESTED ADSORPTION DISTILLATION HYBRID SCHEME

It is our contention that the most practical way to achieve energy savings in the process of propylene separation is to use an adsorption-based process. The adsorption approach allows us to address the problem of propane/propylene separation early in the separation scheme, as shown by the placement of the VSA unit in Fig. 2, i.e., before most of the expensive and energy intensive compression and distillation takes place. The energy spent for compression in the generic scheme (Fig. 1) is almost totally wasted from the point of view of gas-phase adsorptive separation.

For example, a desirable operation for upgrading of refinery-grade propylene to chemical or polymerization-grade product is to use the generic olefin separation scheme as shown in Fig. 1. However, this scheme is a poor proposition if considering the application of gas-phase adsorptive separation because it would involve replacing the C_3 splitters by VSA units; this would require an energy-bearing decompression and warmup of the stream to vaporize the components.

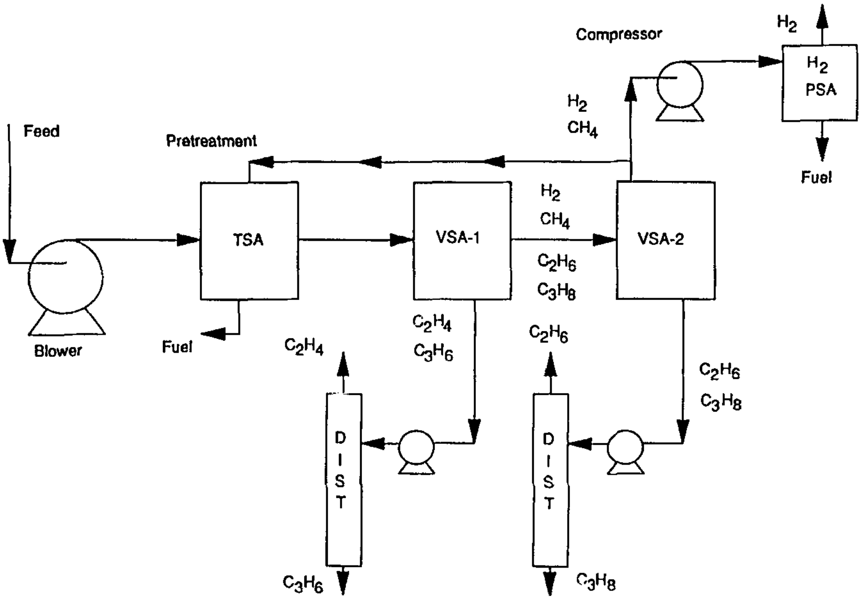

FIG. 2. Hybrid system for propane/propylene separation.

Thus, we are proposing the adsorption–distillation hybrid scheme shown in Fig. 2, which has the potential to reduce the energy consumption and capital cost of propane–propylene separation. Our basic concept is to separate olefins from alkanes by adsorption and then separate individual olefins and alkanes by simple distillation. Therefore, energy intensive and expensive olefin–alkane distillation is eliminated. A conceptual flow schematic for the proposed hybrid scheme is shown in Fig. 2.

In this scheme, feed gas is from a source such as a naphtha-based ethylene cracker. If necessary, the feed gas pressure can be increased to about 15–50 psig by addition of a blower. This feed gas stream then enters an adsorptive pretreatment system where undesired impurities such as heavy hydrocarbons and acetylene are removed by a standard thermal swing adsorption (TSA) process. These impurities, present at trace levels in the feed gas, exit the TSA system during the hot regeneration step. Regeneration gas for TSA is obtained from the second vacuum swing adsorption (VSA-2) system. The TSA system should be designed to minimize propane/propylene loss during the regeneration step. An effluent gas stream during the feed step from the TSA system consists primarily of H_2, CH_4, C_2H_4, C_2H_6, C_3H_6, and C_3H_8.

TSA effluent is further fractionated by the first vacuum swing adsorption system, VSA-1. This process produces two streams. The primary product stream produced at ambient pressure during the evacuation step consists of C_2H_4 and C_3H_6. This stream is sent to a distillation column to produce high-purity ethylene and propylene streams. The secondary product stream from VSA-1 is produced at feed pressure during the adsorption step and primarily consists of H_2, CH_4, C_2H_6, and C_3H_8.

The secondary product stream from VSA-1 is fed to another vacuum swing adsorption process, VSA-2. This process also produces two effluent streams. The primary product stream produced at ambient pressure during the evacuation step consists of C_2H_6 and C_3H_8. This stream is sent to a distillation column to produce high-purity ethane and propane streams. The secondary product stream from VSA-2 is produced at feed pressure during the adsorption step and primarily consists of H_2 and CH_4. Part of this secondary product stream is used to repressurize VSA-1 and VSA-2 beds and regenerate the TSA beds. The remainder of the secondary product stream from VSA-2, if desired, is compressed and fed to a standard pressure swing adsorption (PSA) unit to produce high-purity hydrogen.

PRETREATMENT SYSTEM

The thermal swing adsorption (TSA) pretreatment system consists of two beds undergoing the five steps to continuously process the feed gas and provide a continuous effluent stream, free of undesired impurities to VSA-1.

Typical adsorbent(s) required for the suggested TSA process includes commercially available activated carbon(s). A process similar to the suggested TSA is used to pretreat landfill gas (*13*, *14*) and air before its cryogenic distillation (*15*, *16*).

VACUUM SWING ADSORPTION SYSTEMS (VSA-1 AND VSA-2)

Both the vacuum swing adsorption systems operate in a similar manner and recover the most strongly adsorbed species at ambient pressure. For VSA-1 these species are primarily propylene and ethylene, and for VSA-2, propane and ethane. Secondary effluent from VSA-1 is further processed in VSA-2, and secondary effluent from VSA-2 is further processed in H_2 PSA to recover hydrogen. Both processes require four beds for continuous operation. Corresponding flow schematics for both the processes in the functional mode are shown in Figs. 3 and 4.

Duration of each of the five process steps is from 1–10 minutes. These processes, developed at Air Products, are capable of producing an evac-

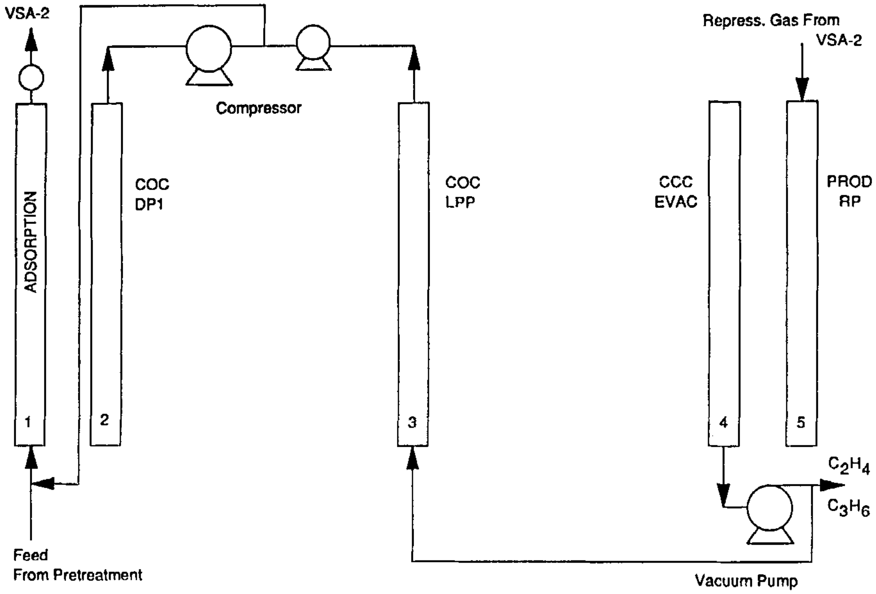

FIG. 3. Functional process description for 4-bed VSA-1.

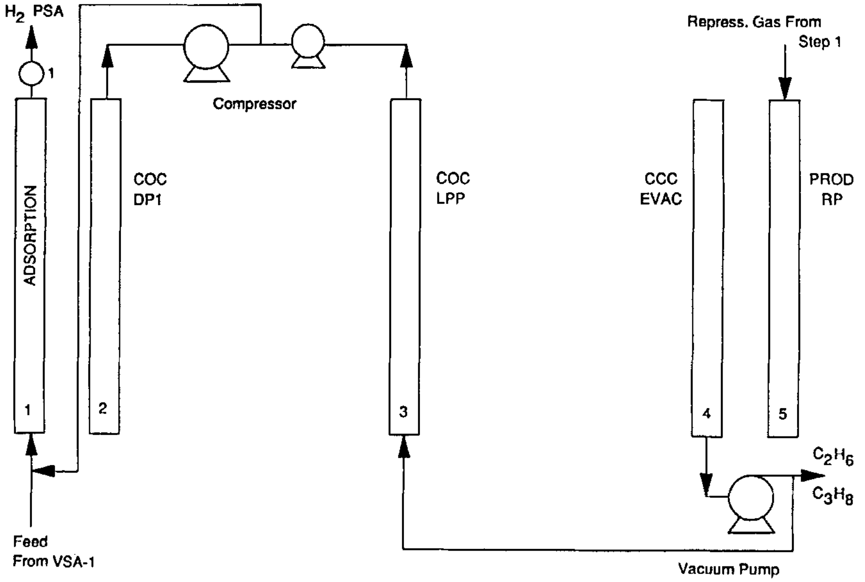

FIG. 4. Functional process description for 4-bed VSA-2.

uated product with both high recovery and purity. Such vacuum swing adsorption processes have been used in the past to obtain carbon dioxide from landfill gas (*14*, *17*), carbon dioxide from SMR off-gas (*18–20*), and other strongly adsorbed species from a variety of gas mixtures (*21*, *22*).

PRESSURE SWING ADSORPTION PROCESS FOR HYDROGEN, H_2 PSA

Secondary effluent from VSA-2 is primarily used for regenerating the pretreatment TSA system and repressurizing the VSA-1 and VSA-2 systems. The remainder of the secondary effluent gas may be further fractionated to obtain high-purity hydrogen in a typical four-bed system. Typical step time for this process is 2–10 minutes.

Adsorbents required for H_2 PSA are commercially available 5A, 13X, or other cation forms of zeolites (*23*) and activated carbons. A large number (>200) of such processes are being used in various industries at the present time.

Depending upon feed composition, feed conditions, product specification, and adsorbent characteristics, several variations of the suggested HYBRID scheme are possible:

If the feed stream is available at moderate pressure, the feed blower may not be necessary.

If the feed stream is clean of undesired impurities, TSA system may not be necessary.

On the other hand, if the feed stream contains impurities which need to be recovered, a separation system prior to VSA-1 or TSA may be required.

If propylene purity from VSA-1 is acceptable, then the olefin distillation column is not needed.

If propane purity from VSA-2 is acceptable, then the alkane distillation column is not needed.

If high-purity hydrogen is not required, then H_2 PSA is not necessary.

If high pressure effluent stream from VSA-1 need not be further fractionated, then VSA-2 is not required.

Therefore, depending upon end-user requirements, the suggested HYBRID scheme configuration may be somewhat different than shown in Fig. 2. However, the central concept of the suggested scheme is paraffin/olefin separation by adsorption (VSA-1 and VSA-2) followed by distillation.

ADSORBENTS

The proposed adsorption scheme for propane/propylene separation requires an adsorbent with both high selectivity and high PSA/VSA working capacities for propylene. A survey of the literature shows that zeolites demonstrate equilibrium selectivity for olefins over alkanes (*24*). This adsorptive selectivity for olefins arises because of a specific interaction between the polar zeolite surfaces and the quadrupole moment of the olefin. However, due to the microporous and acidic nature of zeolites, they 1) adsorb significant quantities of undesired alkanes, 2) have small PSA/VSA working capacities at ambient temperatures because of their rectangular isotherm shape, and 3) are chemically reactive with respect to olefins. Peterson and coworkers (*25*) recognized these problems and proposed a vicinal exchange sorption process using 5A zeolite modified by the preadsorption of ammonia for this separation. Regeneration of the sorbent in this process is achieved by sorption exchange where the sorbed compound (propylene) is eluted by a suitably chosen exchange fluid (pentane). The driving force for adsorption then is sorption exchange, and heating or depressurization are unnecessary. The authors submit that their process offers the advantage of being operable at lower temperature than that required for vacuum regeneration. In addition, the chemical reactivity problem is addressed by decreasing the surface acidity of 5A by preadsorbing ammonia.

However, to our best knowledge there has not been a commercial demonstration of propane/propylene separation using zeolites due to the above-mentioned problems. More recent literature shows that supported CuCl adsorbents demonstrate olefin/alkane selectivity and may be useful for this separation. Hirai (*26*) has shown that an adsorbent selective for ethylene and propylene over their corresponding alkanes can be prepared from a macroreticular polystyrene resin with primary and secondary amino groups and CuCl. The amount of adsorbed olefin increases with an increasing amount of supported CuCl. On the other hand, the amount of alkane adsorbed decreases with an increasing amount of CuCl. This adsorbent adsorbs olefins by coordination of olefins to the Cu(I) ions in the complexes between CuCl and the amino groups of the macroreticular resin. The reported propylene adsorption capacity at 1 atm and 20°C is about 1 mmol/g, and the ratio of propylene to propane capacity at the above conditions (based on pure component isotherms) is 3.5. Both of these values are high enough to suggest that these types of adsorbents may be useful in the proposed VSA scheme.

An adsorbent composition patented by Xie and coworkers (*27*) could also be useful for the proposed separation. The adsorbent consists of a

Cu(I) compound supported on a high surface area support. The addition of Cu(I) salts to the support is achieved by comixing the support and the copper salt and heating the mixture to disperse the salt onto the support. As in the case of Hirai's adsorbent, the selectivity for unsaturated hydrocarbons arise from coordination of the olefin to the Cu(I) ions.

These types of Cu(I)-based adsorbents may well provide the selectivity and capacity required to a VSA separation of propane and propylene without the need for high temperature operation and chemical reactivity of zeolite adsorbents.

ECONOMIC AND ENERGY ANALYSIS

A preliminary economic analysis of the hybrid process scheme is presented which compares the potential economic advantages with a conventional system. The case chosen for this economic analysis is a naphtha-based ethylene plant. The purification section for this plant is expected to be very similar to the olefin purification section of a refinery. Since essentially all propylene is recovered from refinery off-gas and steam cracking of hydrocarbons, this case is representative of actual market conditions for propylene recovery in the United States.

Preliminary research has indicated that the proposed VSA/distillation hybrid system is technically feasible. However, no actual VSA operating data are available for the olefin/paraffin separation. Based upon our prior operating experience with other VSA systems, a typical performance is shown in Table 1. For this analysis it is assumed that the olefin/paraffin VSA unit will perform at the level shown in Table 1.

The base case for this study is a 1000-MM lb/yr naphtha-based ethylene plant. Capital and operating costs for the conventional system are based on studies (*28–31*) performed by the Stanford Research Institute (SRI). Table 2 shows the capital cost comparison and Table 3 shows the energy requirement comparison. The results indicate that the VSA hybrid offers potential energy savings of 30–50% and potential capital cost savings of 20–30% in the purification section of the plant.

TABLE 1
Expected VSA Performance

Olefin recovery	90–99%
Energy requirements	5–10 kW/TPD
Capital cost	15–30 \$M/tpd[a]

[a] Based on 1000 MM lb/yr. Extrapolated from actual VSA cost data in 0–50 MM lb/yr range.

TABLE 2
Capital Cost Comparison
(Basis: 1000 MM lb/yr naphtha-based ethylene plant)

	Conventional	VSA
Compression/refrigeration	80	50
Purification	55	15
VSA	—	25–45
Total cost, $MM	135	90–110
Relative cost	1.00	0.67–0.81

The overall comparison of our system to a conventional system is shown in Table 4. The VSA system is superior to the conventional system in both capital and energy requirements. A key uncertainty at this point is whether the VSA system can match or surpass the olefin recovery of the conventional system. We believe a recovery of 98+% for the VSA system is a feasible target based on actual VSA performance with other separations.

If the VSA target performance can be reached, Table 4 shows that there is sufficient economic incentive to use the VSA hybrid scheme for olefin recovery in new plants.

There is already a huge installed ethylene/propylene capacity, and retrofitting the VSA scheme into this market would offer enormous potential. Table 5 shows the economic impact of retrofitting the VSA system into

TABLE 3
Potential Energy Savings
(Basis: 1000 MM lb/yr naphtha-based ethylene plant)

	Conventional[a]	VSA
Precooling	400	200
Demethanizer	600	—
De-ethanizer	600	600
C_2 Splitter	2000	—
Depropanizer	350	350
C_3 Splitter	700	—
VSA	—	600–1200[b]
Compression	1275	850
Total energy, Btu/lb C_2H_6	5925	2600–3200
Relative	1.00	0.44–0.54

[a] Based on SRI data in Report 29B for Steam and Refrigeration

[b] Based on 5–10 kW/tpd of olefin and 10,000 Btu per kWh based on using electric power

TABLE 4
Conventional vs VSA Comparison

	Conventional	VSA[a]
Olefin recovery	98+%	90–99%
Relative capital	1.00	0.70–0.85
Relative energy	1.00	0.45–0.55

[a] Data is based on Air Products experience on other separations using VSA systems.

ethylene plants. A best and worst case is presented for two options. All new equipment is used in Option A, while in Option B the existing compression and distillation equipment are used with a new VSA system.

The results for the retrofit case (Table 5) indicate that using all new equipment (Option A) is not an economically viable alternative. However, the results also indicate that there is a potential opportunity if existing equipment can be used (Option B). The capital cost of the VSA system and the olefin recovery levels of the VSA system will be the key factors that establish the viability of the VSA system for retrofit cases.

Table 5
Retrofit VSA Cases
(Basis: 1000 MM lb/yr naphtha-based ethylene plant)

	VSA Option A		VSA Option B[a]	
	Best	Worst	Best	Worst
A) Capital cost:				
Compression, refrigeration	50	50	—	—
Purification	15	15	—	—
VSA	25	45	25	45
Total, $MM	90	110	25	45
B) VSA energy saving Btu/lb C_2H_6	3300	2700	3300	2700
C) Retrofit savings:				
Capital charges at 35%	(2.8)	(3.5)	(0.9)	(1.6)
Energy saving at $4/MM Btu	1.3	1.1	1.3	1.1
Net savings, ¢/lb	(1.5)	(2.4)	0.4	(0.5)
Net savings, $MM/yr	—	—	4.0	—

[a] Based on using existing equipment.

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