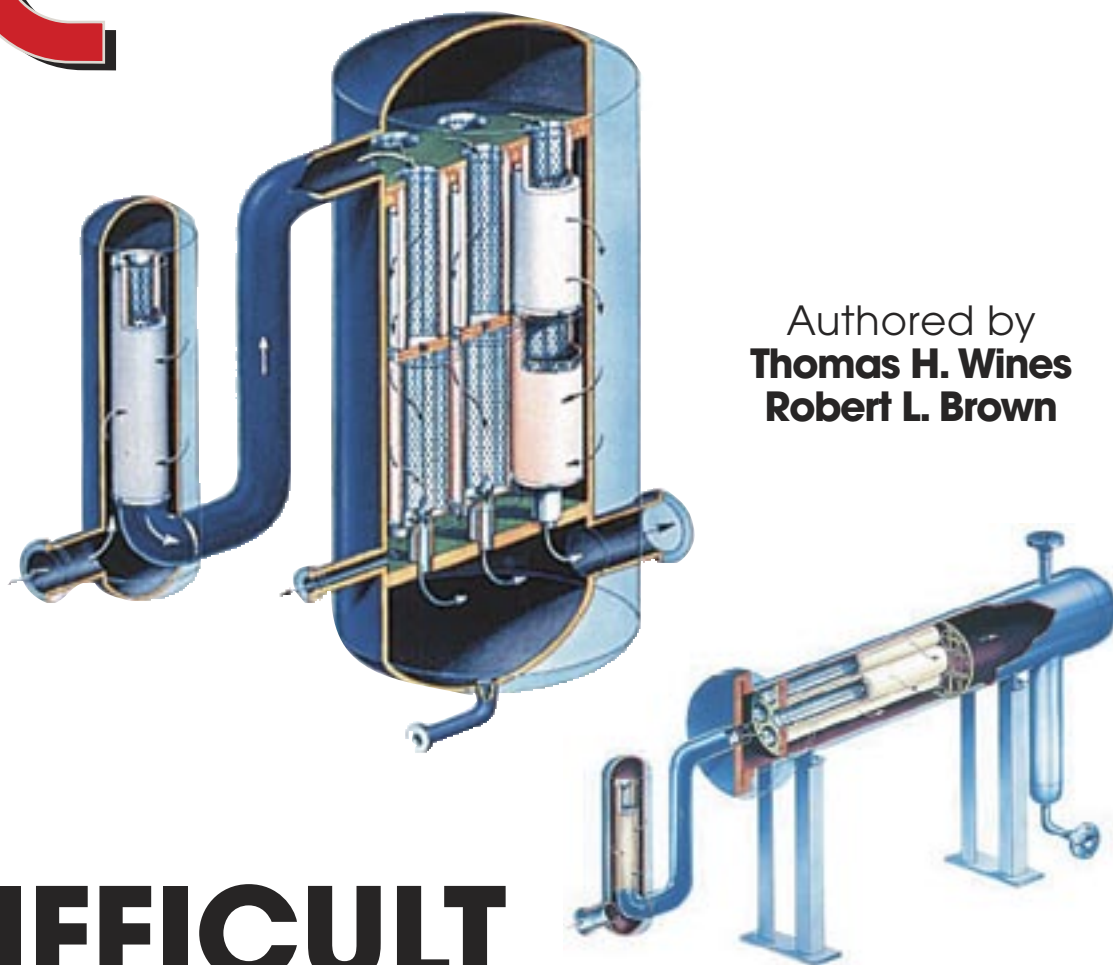


# CHEMICAL ENGINEERING

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Authored by  
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## DIFFICULT LIQUID-LIQUID SEPARATIONS

**PALL** Pall Corporation

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# DIFFICULT LIQUID-

## High-performance, polymer-fiber coalescers break up hard-to-handle emulsions and dispersions

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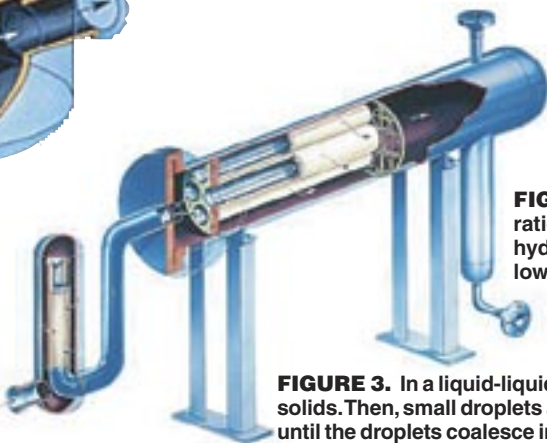
Common sources of contamination in chemical and petrochemical processes are liquid dispersions and emulsions formed during liquid-liquid extraction. These often happen where mixing of solvent and feed is needed to optimize extraction efficiency, but results in solvent carryover into an extract or raffinate. Large amounts of solvent can be carried over into product streams in process units running at rates above design capacity, for example.

Carried-over liquid causes off-specification products and excessive solvent losses, and, often, operating problems in downstream processes. If water is present in process fluid, it can form stable emulsions that can cause off-specification products.

Dispersions and emulsions are often treated by coalescing, in which immiscible liquids are divided using the liquids' interfacial tension and their different rates of wetting on a porous medium. Traditional glass-fiber coalescers, however, cannot meet the challenges of difficult-to-separate fluids, such as those containing large amounts of surfactants.

Cartridge coalescers using new polymer-based fibers now offer enhanced performance and higher productivity. Unlike glass, polymeric materials can be processed into fine fibers of varying diameters with tapered pore structures and improved surface properties.

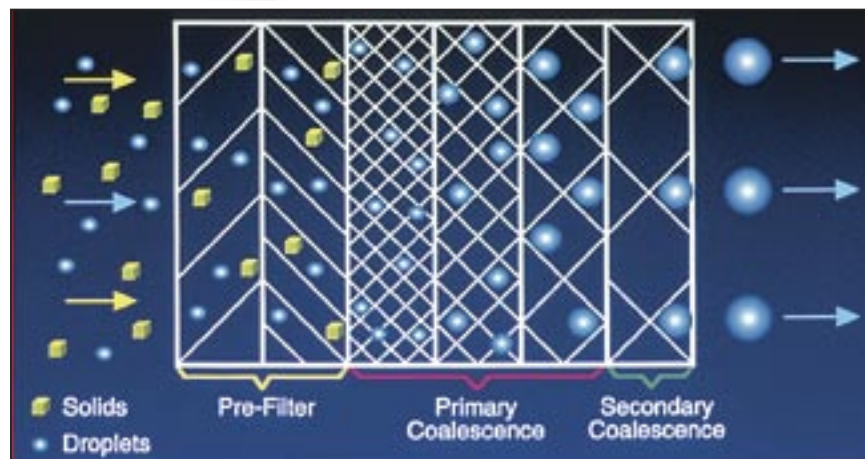
This article discusses the basic configurations and operation of a high-performance coalescer. To understand the technology, it is also necessary to understand the physical mechanisms of



**FIGURE 1.** A vertical configuration (far left) of a liquid-liquid coalescer is for removing aqueous materials from a hydrocarbon fluid

**FIGURE 2.** This horizontal configuration (left) removes aqueous fluids from hydrocarbons when interfacial tension is low — below 3 dynes/cm

**FIGURE 3.** In a liquid-liquid coalescer, a prefilter first removes solids. Then, small droplets are combined into larger droplets, until the droplets coalesce into a bulk-flow fluid



coalescence, including interfacial tension, concentration of surface-active compounds, steric repulsion and electrostatic-charge effects on particles. These concepts are discussed in later sections of the article.

**Terminology.** The term “hydrocarbon” is used in this article to describe the nonpolar phase, which can include various organic chemicals with low polarity, such as long-chained alcohols and methylene chloride. The term “aqueous” refers to the polar phase, which, in addition to water and dissolved inorganic salts, can also comprise polar chemicals, such as short-chain alcohols and ammonia.

### Stages of a coalescer system

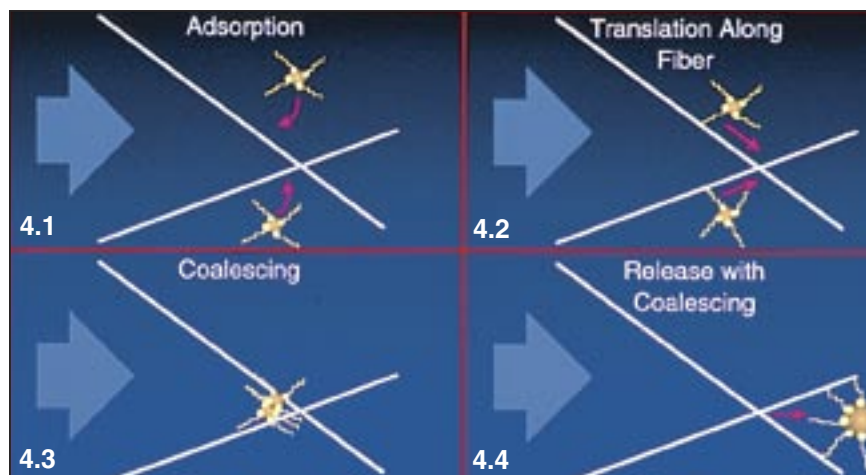
The liquid-liquid coalescer system consists of three stages: prefilter, coalescer and separator. The prefilter stage is generally a separate vessel with filter cartridges. The coalescing and separator stages can be configured vertically or horizontally.

### Vertical configuration

This setup (Figure 1) is for removing an aqueous dispersed contaminant from a continuous hydrocarbon fluid. It has been used to separate aqueous-hydrocarbon systems with interfacial tensions as low as 3 dynes/cm.

Coalescer cartridges are stacked on

# LIQUID SEPARATIONS



**FIGURE 4.** Fibers foster the coalescing mechanism by moving droplets to junctures where they join with other droplets to form larger ones

top of hydrophobic barrier-separator cartridges. Process fluid enters the top of the housing and then flows from the inside of the coalescer cartridges radially outward, causing the enlargement or coalescing of the inlet dispersion into large droplets in the outlet stream.

The coalesced droplets then flow axially downward with the convective flow, and are repelled by a hydrophobic separator-barrier cartridge. The purified, continuous-phase fluid flows from the outside of the separator cartridges to the inside, and leaves the vessel as a purified stream. The coalesced aqueous droplets are collected in a sump section at the bottom of the housing.

## Horizontal configuration

This setup (Figure 2) is used for the removal of an aqueous dispersed contaminant from a bulk hydrocarbon stream when the interfacial tension is below 3 dynes/cm. It can also be used to remove nonaqueous dispersed contaminant from a continuous aqueous stream. The system consists of a horizontal coalescer-cartridge stage, followed by a settling zone that uses the phases' difference in density to separate the coalesced droplets.

In this configuration, fluid enters at the side of the housing and flows from the inside of the coalescer cartridges ra-

dially outward, causing the enlargement or coalescing of the inlet dispersion into large droplets in the outlet stream. The coalesced droplets flow axially in the horizontal direction through a settling zone.

If the specific gravity of the dispersed phase is greater than that of the continuous phase (i.e., aqueous-from-hydrocarbon separation), the coalesced droplets settle downward by gravity and are collected in a sump at the bottom of the housing. If the specific gravity of the dispersed phase is less than that of the continuous phase (i.e., hydrocarbon-from-aqueous separation), the coalesced droplets are collected at the top of the housing.

**Solids filtration.** Solids, which may increase the stability of an emulsion, can be removed prior to coalescing. Generally, this step is achieved via a separate cartridge-filter system, or a regenerable backwash-filter system for high levels of solids. This filtration stage also protects the coalescer and increases service life.

## Coalescence

After filtration, the next step is the primary coalescence. In this stage, the coalescer's pore dimensions begin with a very fine structure, gradually opening to allow for void space for the coalescing

droplets (Figure 3). In the primary coalescence zone, the inlet droplet dispersion containing fine droplets (0.2–50 micrometers) is transformed into a suspension of enlarged droplets (500–5,000 micrometers).

When a fibrous medium is used, the coalescence mechanism is described by the following steps (Figure 4):

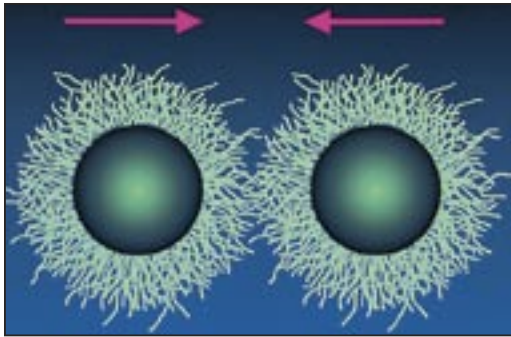
1. Droplet adsorption on fiber
2. Translation (movement) of droplets to fiber intersections by bulk flow
3. Coalescence of two droplets to form one larger droplet, and repeated coalescence of small droplets into larger droplets at fiber intersections
4. Release of droplets from fiber intersections due to increased drag on adsorbed droplets, caused by bulk flow
5. Repeat of steps 1–4 with progressively larger droplet sizes and more-open media porosity

Based on this mechanism, we can predict that a number of factors will influence the coalescence performance. The specific surface properties of the coalescer fibers are critical in the adsorption of droplets, as well as the release of droplets after coalescing. There is a balancing act between the attraction (adsorption) characteristics of the fibers versus the release mechanism, which would be inhibited by strong adsorption [1, 2].

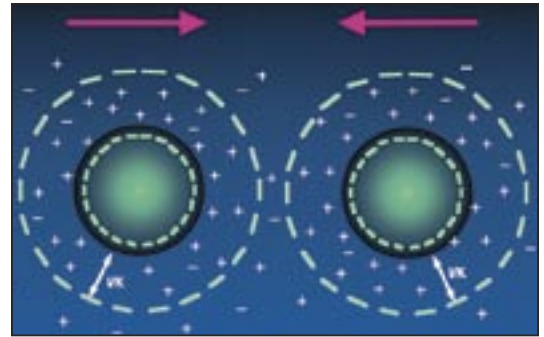
**Surfactants' detrimental effect.** Added to water in hydrocarbon systems, surfactants generally decrease the interfacial tension between phases. (Interfacial tension, or the energy per unit area at the boundary of two immiscible liquids, is discussed further in a later section).

Decreased interfacial tension, in turn, generally causes coalescers to "disarm," or become deactivated. This disarming effect occurs when surfactants bond with the adsorption sites on the coalescer, shielding them from incoming water droplets. Unable to adsorb onto the coalescer's surfaces, the droplets thus are not able to coalesce with other droplets.

In most cases, an interfacial tension



**FIGURE 5.** Surfactant molecules surround a micelle or reverse micelle with their nonpolar ‘tails’ facing outwards (left), which repel other micelles through steric repulsion. This differs from electrostatic repulsion (right), where a charged particle inside a micelle creates the repelling field



of less than 20 dynes/cm causes disarming in glass-fiber coalescers. Polymeric media, however, can be surface modified to prevent disarming.

The explanation for the modified polymer fibers’ resistance to disarming is that there is less of an attraction for the surfactants’ polar heads to adsorb onto the fibers. Due to the enhanced surface properties of the new coalescer medium, the surfactants actually adsorb by the nonpolar tails.

The polar heads face outward, attracting passing water droplets. The coalescer medium retains the open sites needed for water droplets to adhere and coalesce normally.

**Separation of coalesced droplets**

Once the droplets have coalesced, they are assumed to be as large as possible for the given flow conditions. Estimates for the size of the coalesced droplets can be made using the following equation [3, 4, 5]:

$$d = (\kappa \sigma / \Delta\rho)^n \tag{1}$$

where  $d$  = droplet diameter,  $\sigma$  = interfacial tension,  $\Delta\rho$  = difference in densities between the dispersed and bulk phase,  $We$  = Weber number,  $k$  = constant =  $f(We)$ , and  $n$  = exponent constant. Other correlations have included a viscosity term.

The Weber number is the dimensionless parameter that relates the order of magnitude of the inertial (shear) forces to that of the surface-tension forces. It is given by:

$$We = \rho d V^2 / \sigma \tag{2}$$

where  $V$  is the velocity of the droplet and  $\sigma$  is the interfacial tension. The critical value of the Weber number in liquid-liquid systems has been reported [5, 6] to be 1–2. Above this crit-

ical Weber number, the droplets become unstable and break up into smaller fragments. The Weber number increases with droplet diameter, velocity, and decreasing interfacial tension.

Various values, based on the Weber number, have been postulated for the constant  $k$ . The value of the exponent  $n$ , which is a constant, is reported throughout the literature as 1/2 or 3/5.

The separation stage can be achieved via either a cartridge element with barrier capabilities, or a settling zone that relies on the difference in densities between the coalesced droplets and the bulk fluid. The choice of method is dependent on the physical properties of the two fluids.

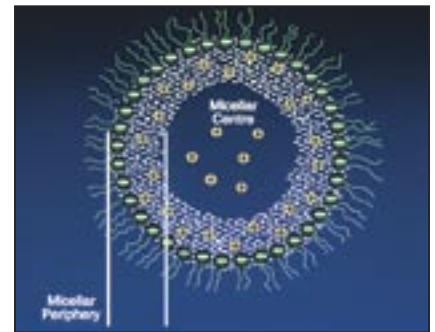
**What makes coalescing difficult**

Stable emulsions generally require the presence of at least three components to form: the two bulk immiscible phases, plus a small concentration of surfactant. Some common sources of surfactants include:

- Corrosion inhibitors
- Organic acids in hydrocarbon feed
- Well-treating chemicals
- Sulfur compounds
- Other chemical additives

An emulsion’s stability can be measured in terms of interfacial tension. Interfacial tension, or surface energy per area, can be expressed in equivalent units of force per distance (usually dynes/cm). Interfacial tension can also be described as the work required to create additional surface area.

The presence of surfactant leads to the formation of micelles (hydrocarbon in aqueous phase — Figure 5) or reverse micelles (aqueous phase in hydrocarbon — Figure 6), depending on the



**FIGURE 6.** Water forms a ‘reverse micelle’ in oil

relative concentrations of the aqueous and hydrocarbon phases.\*

The internal energy of an emulsion system increases proportionally with increasing area and interfacial tension. Lower interfacial tension leads to the formation of more, smaller emulsified droplets, which are harder to coalesce.

Emulsions are stabilized by such mechanisms as steric repulsion, electrostatic charge repulsion and resistance to film drainage. These factors influencing emulsion stability are discussed further below.

**Steric repulsion from surfactants.** Reverse micelles have an inner aqueous phase bounded by a surface layer of surfactant molecules. The surfactant molecules orient themselves so that the polar heads are adsorbed by the aqueous core, while the nonpolar, relatively long-chain sections point radially outward. The nonpolar chain sections sterically repel other micelles that approach too close [7, 8, 9].

The mechanism for spontaneous coa-

\*The formation of micelles in aqueous systems is attributed to London-van der Waal forces between similar hydrocarbon chains and an overall entropic increase when hydrated water molecules are released. Reverse micelles formed in the hydrocarbon phase are attributed to hydrogen bonding between water molecules and polar headgroups of the surfactant.

lescence is depicted for the case of water droplets in a hydrocarbon stream with and without surfactant (Figure 7). When surfactants are not present in a water-hydrocarbon system, coalescing occurs spontaneously (i.e., without a coalescer). Surfactants, however, can create a stable emulsion that cannot be separated without special equipment, such as a high-performance coalescer.

The disarming experienced by glass fiber coalescers [10] indicates that fiber adsorption is central to the coalescing mechanism. Figure 8 illustrates how adsorption leads to disarming.

The surfactant molecules adsorb preferentially to the active sites on the glass coalescer fibers and orient themselves with their nonpolar tails facing outward from the fibers. The dispersed aqueous droplets also have a layer of surfactants in similar orientation, with nonpolar tails facing outward. Coalescence is inhibited by steric repulsion between the adsorbed surfactant on the droplets and the coalescer fibers.

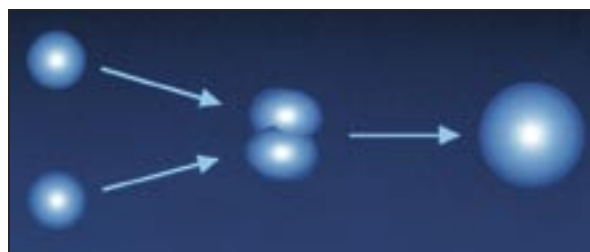
**Charge repulsion from ions, acids and bases.** At the center of micelles, charged species, including water, dissolved ions and particles, generate a balancing electrostatic charge that extends into the continuous phase. Adjacent micelles or reverse micelles with like charges repel each other. Tests of modeling colloids (i.e., oil micelles) in aqueous solutions show that, generally, as continuous-phase conductivity decreases, electrostatic repulsion increases [11], deterring coalescence.

**Film stabilization.** Fine particulate materials, such as iron oxides or iron sulfides, accumulate at the boundaries of the droplets. These particles reinforce the film surrounding the droplets, making it harder for coalescence to occur.

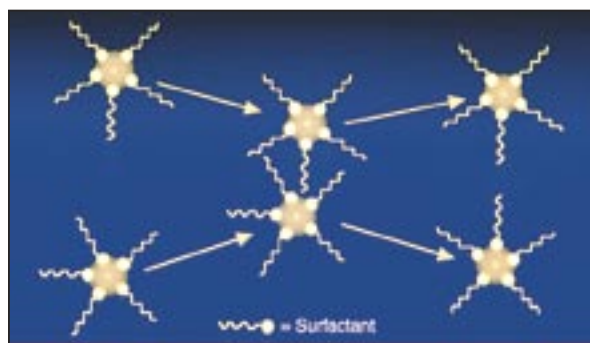
The solid particles must be removed from the film surface before coalescing can take place. Removing the solids with a filter can eliminate some of this added resistance and improve coalescing efficiency.

### Commercial applications

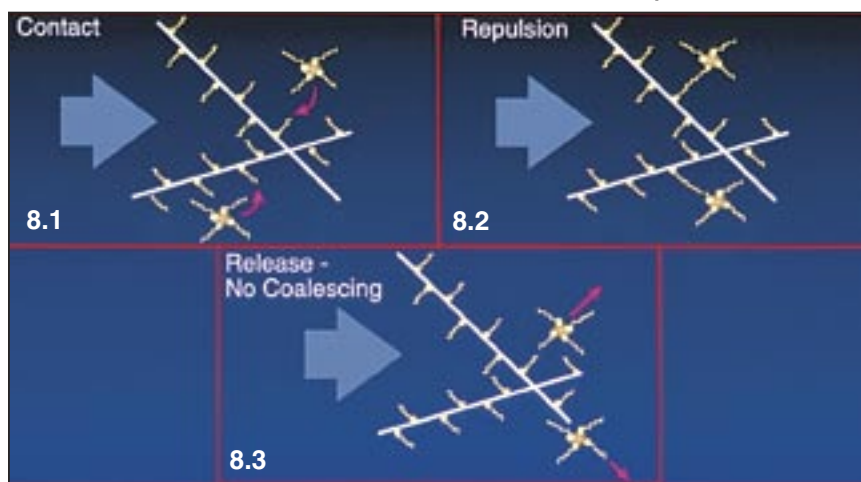
High-efficiency, specially formulated technology for polymeric liquid-liquid coalescing and separations has been tested and is now commercially operating in some gas-processing plants



**FIGURE 7.** Bare water droplets (left, top) spontaneously coalesce in free space, while surfactant-laden droplets (left, bottom) form swollen micelles that repel each other, and are thus harder to remove from a hydrocarbon stream



**FIGURE 8.** Surfactant molecules coat coalescer fibers with their nonpolar tails facing outward (8.1). Other droplets are unable to 'land' on the fibers (8.2), and are thus unable to coalesce (8.3). This phenomenon is called



and refineries. This new method differs from existing commodity liquid-liquid coalescers using glass-fiber medium, which is prone to disarming when interfacial tension is below 20 dynes/cm.

### Agricultural chemicals

**Process description.** In an agricultural chemical plant in Europe, proprietary reactor and catalytic technology is used to make a chemical intermediate, based on an aromatic solvent. This intermediate is later used to make plant-protection products.

In the last step of the process, a water wash removes organic acids, a by-product of the reaction from the final product. Final product is sent to a storage tank and loaded into tank cars.

**Application and experience.** The

product tanks experienced severe corrosion problems due to the presence of acidic water. Because the organic acids are surface-active chemicals, efficient separation of the water-acid mixture from the product could not be accomplished with a knock-out drum. Analysis indicated that the interfacial surface tension was 11 dynes/cm, verifying that the aromatic solvent-acidic water mixture was a stable dispersion.

In addition, the carried-over aqueous material caused operational problems in the downstream process. For example, aqueous contaminant concentrations of 1–2 vol.% in the aromatic solvent necessitated high energy usage when the product was processed in a distillation column. At times, the unit's operating capacity was limited by the

**TABLE 1. REMOVAL OF CAUSTIC FROM HYDROCARBON VIA SAND FILTER AND LIQUID-LIQUID COALESCER**

	High	Low	Average
Inlet concentration of NaOH (mg/L)	69.9	3.5	29.5
Outlet concentration of sand filter (mg/L)	7.6	0.7	4.2
Outlet concentration of liquid-liquid coalescer (mg/L)	0.5	0.0	0.2

distillation-column reboiler.

The company installed a polymeric liquid-liquid coalescer unit to minimize corrosion and improve the efficiency of the downstream process by removing the aqueous contaminant. After eight months of operation, the following improvements have been observed:

- Reduced corrosion in the product-storage tank by eliminating residual acid-free water
- Enhanced efficiency of the distillation column in the downstream process
- Improved condenser maintenance on top of the distillation column in the downstream process
- Increased capacity in the downstream process

### Mixed butanes and gasoline

**Process description.** Caustic treating is used to either remove mercaptans from fuels, or convert them to more-desirable disulfides. The reaction takes place in the presence of air and caustic in a fixed catalyst bed or a contactor.

A settling drum separates the product from the caustic, which is subsequently recirculated to the front end of the treating unit. Problems typically occur when there is poor separation in the settling drum, and caustic carries over into the product.

**Application and experience.** A polymeric liquid-liquid coalescer that is compatible with caustic is used to remove carried-over caustic. In field tests conducted on a light hydrocarbon stream, the coalescer performance, in terms of removing sodium hydroxide, was compared to a sand filter. These results are summarized in Table 1.

Analysis of the sodium hydroxide was performed using a titration method. Results are based on 13 samples taken over a 3-week period. Currently there are five operating units worldwide in gas plants and refineries using the new polymeric liquid-liquid coalescer technology to remove carried-over caustic from hydrocarbon.

An analysis of the interfacial tension explains why the sand filter was not effective in removing the caustic. The interfacial tension analyses ranged from less than 1 dyne/cm to 12 dynes/cm, which is below the recommended limit of glass coalescers.

**TABLE 2. PROCESS PARAMETERS FOR CAUSTIC RECOVERY VIA LIQUID-LIQUID COALESCER**

Fluid	Gasoline, caustic
Flowrate	20,100 bbl/d
Interfacial tension	12 dynes/cm
Inlet water, caustic	2,250 parts per million by volume, ppm <sub>v</sub> (total)
Caustic strength	2.3 wt.%
Outlet water	110 ppm <sub>v</sub> (total)
Projected annual caustic savings	126,000 lb/yr

**TABLE 3. PROJECTED ECONOMICS OF AMINE RECOVERY WITH LIQUID-LIQUID COALESCER**

Flowrate	250 gal/min
Total losses	120 gal/d
Specific gravity of LPG	0.488
Specific gravity of amine	1.05
Concentration of amine	40 wt.% in water
Total losses	333.3 parts per million by volume (ppm <sub>v</sub> ), 717.2 ppm by weight (ppm <sub>w</sub> )
Solubility of amine in LPG	160.0 ppm <sub>w</sub>
Losses expected from carryover	60%
Expected recovery of amine	319.3 ppm <sub>w</sub> 148.4 ppm <sub>v</sub> 53.4 gal/d 19,501 gal/yr 170,773 lb/yr
Recovery of total solution	134 gal/d 48,753 gal/yr
Cost of amine	\$0.50/lb
Estimated savings	\$85,386/yr

This low interfacial tension formed a difficult-to-separate, stable emulsion. Surface-active sulfur compounds in the spent caustic probably contributed to the extremely low interfacial tension.

A polymeric liquid-liquid coalescer recently installed on a refinery gasoline system provided caustic savings of 126,000 lb/yr (Table 2). Although the projected caustic savings alone provided a payback in less than a year, greater benefits were realized from a decrease in off-specification "hazy" gasoline and the reduced corrosion of downstream piping and tankage.

### Amine treating of LPG

**Process description.** Amine treating is for removing hydrogen sulfide or

carbon dioxide from light liquid hydrocarbons (liquid petroleum gas; LPG). The reaction takes place in a liquid-liquid contactor tower, where hydrocarbon feed is introduced at the tower bottom through a distributor. Amine treating solution is introduced countercurrently at the top of the tower.

The treated product is removed from the top of the tower, while the contaminant-laden amine solution leaves from the tower bottom and is sent for thermal regeneration.

Typically, a knockout drum is located downstream of the contactor tower overhead to recover any carried-over amine. Problems occur when there is poor separation in the knockout drum, and amine carries over into the product.

### Application and experience.

Field testing was conducted with a new polymeric liquid-liquid coalescer on this application in three locations. The concentration of the amine downstream of the polymeric liquid-liquid coalescer was found to be roughly the same as the solubility of the amine in LPG. In one location, the amount of amine that coalesced out instantaneously was three times the amount that settled out in a tank over one full day.

Carried-over amine is a major contributor to amine losses. If recovered via coalescence, amine can be recycled back to the contactor. Projected economics for a recently evaluated system at a gas-processing plant are summarized (Table 3).

Here, the expected payback is again less than a year. This economic analysis takes into account only recovery of amine losses. Even more savings are realized by decreased downstream corrosion and equipment fouling, and increased product quality.

Due to the presence of sulfur compounds, the interfacial tension in this particular case is again expected to be low. Note that, because LPG flashes when it is brought to atmospheric pressure, it is difficult to perform an accurate analysis of interfacial tension in this case.

TABLE 4. NEW COALESCER APPLICATIONS TESTED IN THE CPI

Dispersed phase	Continuous phase	Result	Competitive technology
Potassium hydroxide solution	Propane	Successful	Settling tank
Sodium hydroxide	Gasoline	Successful	Settling tank
Water	Dicumyl peroxide	Successful	Sand filter, electrostatic precipitator
Water	Phenol, acetone, cumene	Successful	Settling tank
Lube oil	Ammonia	Product modification	Glass coalescer
Water	Herbicide	Successful	Settling tank
Water	Dihydro-peroxide	Successful	Vane separator
Water	Ethers	Successful	Settling tank
Water	Methyl siloxane	Successful	New application
Methane sulfonic acid solution	Methacrylate	Successful	Settling tank

TABLE 5. COMPETING TECHNOLOGIES USED IN CPI INSTALLATIONS

Dispersed phase	Continuous phase	Competitive technologies
Water	Oxo-alcohols	Desiccants
Organics	Crude peroxide	Stainless steel mesh coalescer
Mineral oil	Sodium borohydride	Settling tank
Water	Styrene	Settling tank
Hydrogen peroxide	Working solution	Glass-fiber coalescer
Water, methanol	Toluene, cyclopropyl butadiene	Settling tank
Water	Toluene, styrene, acrylonitrile	Settling tank
Silicone oil	DMAC-water mix	Settling tank
Water	Liquid organic peroxide	Glass-fiber coalescer
Water	Mixed aromatics	Settling tank
Water	Butyl acetate	Settling tank

TABLE 6. PETROLEUM-REFINERY USES AND COMPETING METHODS

Application	Dispersed phase	Continuous phase	Competing technology
Refined fuels	Water	Gasoline, diesel, jet fuel, LPG	Settling tanks, glass-fiber coalescers
Clay treater protection	Water	Kerosene	Vacuum dehydration, settling tanks, nothing
Salt-dryer protection	Water	Hydrocarbons	No protection
Mercox treater	Caustic	Hydrocarbons	Sand filters, settling tanks, electrostatic precipitators
Glycol recovery	Glycol (TEG)	natural gas liquid	Mist eliminators, knockout drums
Methanol recovery	Water, methanol	Liquid petroleum gas	Distillation

TABLE 7. POLYMERS USED FOR COALESCERS

Polymer type	Typical compatible fluids*
Polypropylene	Acids, bases, alcohols, ammonia, amines
Polyethylene	Acids, bases, ammonia, amines
Polyester	Water, hydrocarbons
Polyamide	Bases, hydrocarbons
Polysulfone	Bases, hydrocarbons
Aramid	Water, hydrocarbons (high temperature)
Polyvinylidene fluoride	Acids, bases, hydrocarbons, alcohols, amines
Polytetrafluoro ethylene	Acids, bases, hydrocarbons, alcohols, ketones, peroxides
Polyphenylene sulfide	Acids, bases, hydrocarbons, ketones, amines

\*Note: Selection of a polymeric coalescer alone does not guarantee performance; the polymer must be processed and shaped specifically for the required coalescing properties

**Other applications**

Field trials have been conducted to test the efficiency of new polymeric liquid-liquid coalescers in several CPI applications (Table 4). In all cases, the new technology equals or exceeds the performance of the existing separation

equipment. This is usually because the interfacial tension is lower than 20-25 dyne/cm. New installations for chemical processes (Table 5) and operating systems for petroleum-refinery applications (Table 6) are listed.

*Edited by Irene Kim*

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