



## Minimizing Liquid Contaminants in Natural Gas Liquids

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In processing natural gas liquids, significant contamination occurs with liquid dispersions and emulsions. Natural gas liquids (NGL) and liquid petroleum gas (LPG) streams are treated with caustic to remove residual organic sulfur compounds such as mercaptans and with amines to remove hydrogen sulfide. In both cases a liquid/liquid contactor is used. Significant amounts of the caustic or amine can be carried over into the product stream in process units that are running at rates above design capacity, are treating high sulfur feed stocks, or have other operational problems. The carried over liquid results in off-spec products, excessive losses of caustic or amine, and can cause operating problems in downstream processes. In addition, water is a significant contaminant which can cause LPG and natural gasoline to be off-specification.

This paper will discuss a new technique for separating very stable liquid dispersions of caustic, amine, or water from natural gas liquids using liquid/liquid cartridge coalescers (L/L) constructed with specially formulated polymer medium with enhanced surface properties. In addition, factors influencing the coalescer mechanism will be discussed including interfacial tension, concentration of surface active compounds, steric repulsion, and electrostatic charge effects. Results from field tests, operating data from commercial installations, and economic benefits will also be presented.

- ▶ [Description of Liquid/Liquid Coalescer System](#)
- ▶ [Solid Filtration](#)
- ▶ [Coalescence](#)
- ▶ [Separation of Coalesced Droplets](#)
- ▶ [Molecular Forces Controlling Coalescence](#)
- ▶ [Steric Repulsion](#)
- ▶ [Electrostatic Charge Repulsions](#)
- ▶ [Film Stabilization](#)
- ▶ [Overcoming Energy Barriers - Coalescer Mechanisms](#)
- ▶ [Commercial Application of Liquid/Liquid Coalescers](#)
- ▶ [Conclusions](#)
- ▶ [References](#)

### Description of Liquid/Liquid Coalescer System

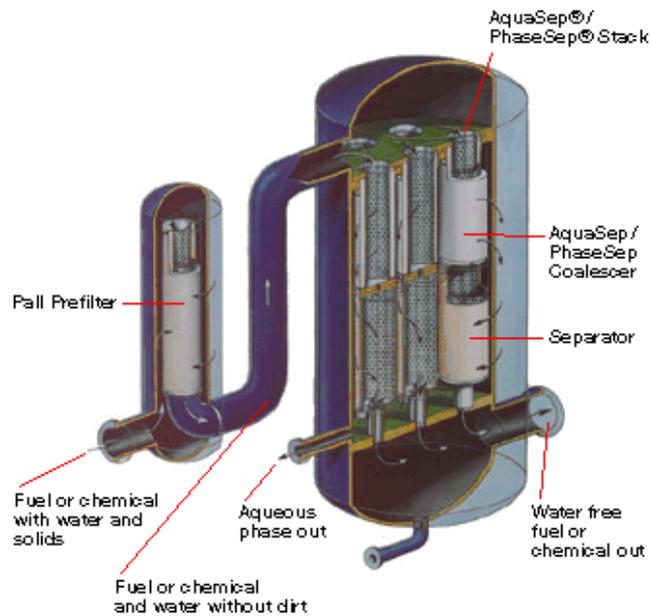
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The liquid/liquid coalescer system consists of three stages: prefilter, coalescer, and separator. The prefilter stage is generally a separate vessel with absolute rated filter cartridges. The coalescing/separator stages can be configured in two possible ways:

#### Vertical Configuration

The vertical configuration (Figure 1) is used for the removal of an aqueous dispersed contaminant from a continuous hydrocarbon fluid. This configuration has separated aqueous-hydrocarbon systems with interfacial tensions as low as 3 dyne/cm. The system contains coalescer cartridges stacked on top of hydrophobic barrier separator cartridges. The 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.

**Figure 1:** Vertical Liquid / Liquid Coalescer Configuration

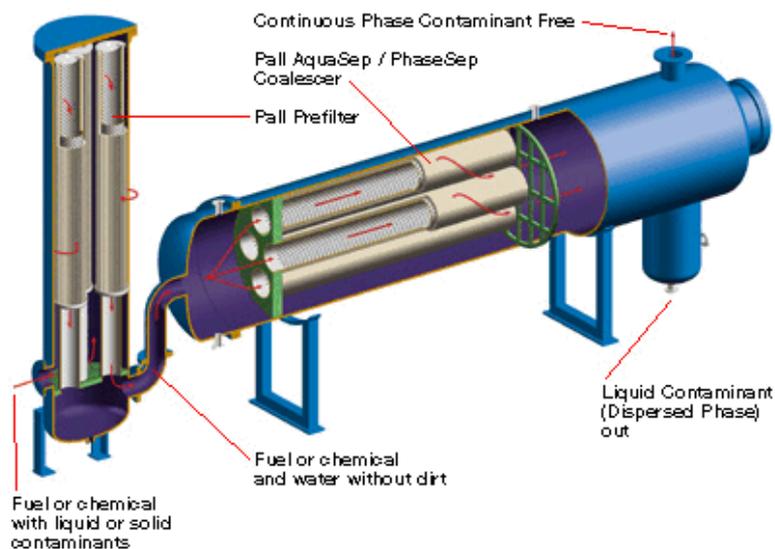


The coalesced droplets then flow axially downward with the convective flow and are repelled by a hydrophobic separator barrier cartridge. The purified bulk hydrocarbon fluid then flows from the outside of the separator cartridges to the inside and leaves the vessel as a purified stream. The coalesced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

### Horizontal Configuration

The horizontal configuration (Figure 2) is used for the removal of an aqueous dispersed contaminant from a bulk hydrocarbon stream when the interfacial tension is below 3 dyne/cm and can be used to remove non-aqueous dispersed contaminant from a continuous aqueous stream. The system consists of a horizontal coalescer cartridge stage followed by a settling zone that relies on the difference in density for separation of the coalesced droplets. The fluid enters at the side of the housing and 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 in the horizontal direction through a settling zone. If the specific gravity of the dispersed phase is greater than the specific gravity of the continuous phase (water from oil separation), the coalesced droplets settle downward by gravity and are collected in a sump located at the bottom of the housing. If the specific gravity of the dispersed phase is less than the specific gravity of the continuous phase (oil from water separation), the coalesced droplets are collected in a sump located at the top of the housing.

**Figure 2:** Horizontal Liquid / Liquid Coalescer Configuration



[Top](#)

### Solid Filtration

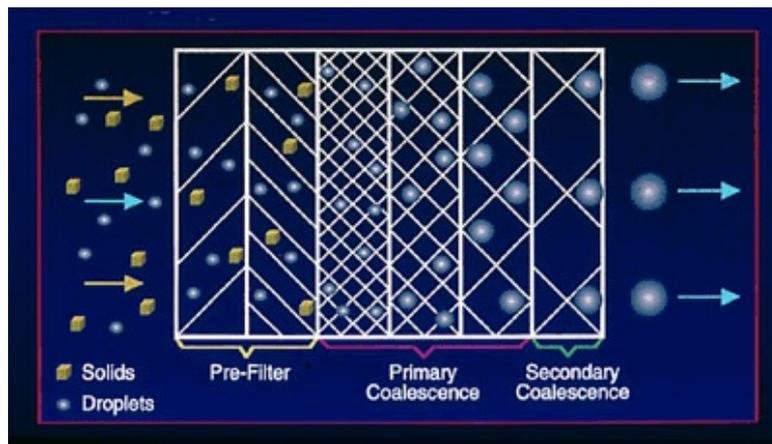
This step can be achieved by a separate cartridge filter system or by a regenerable backwash filter system for high levels of solids. The purpose of the filtration stage is to protect the coalescer and the type of filter is selected on an economic basis.

[Top](#)

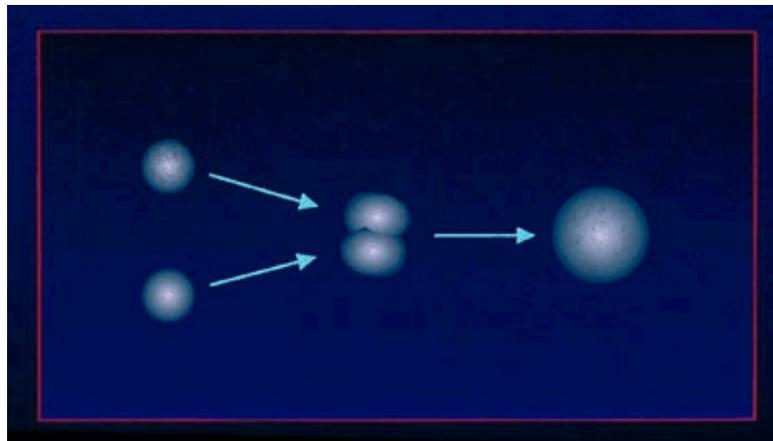
## Coalescence

The next step in the process is the primary coalescence. In this stage, the pore structure begins with a very fine structure and then becomes more open to allow for void space for the coalescing droplets (see Figure 3). In the primary coalescence zone, the inlet droplet dispersion containing fine droplets in the size range of 0.2 to 50 microns is transformed into a suspension of enlarged droplets in the size range of 500 to 5000 microns. The state of the suspended water in the hydrocarbon prior to passing through a coalescing medium warrants further discussion. In Figure 4, the mechanism for spontaneous coalescence is depicted for the case of water droplets in a hydrocarbon stream with and without surfactant. When surfactants are not present in a water-hydrocarbon system, coalescing will occur spontaneously without the need for fibrous medium to aid in the process. When surfactants are present, the result may be a stable emulsion which cannot be separated unless special steps are taken such as the use of high performance liquid/liquid coalescer cartridges. Water in hydrocarbon emulsions are stabilized by such factors as steric repulsion, electrostatic charge repulsion and resistance to film drainage. These factors will be discussed further in this article.

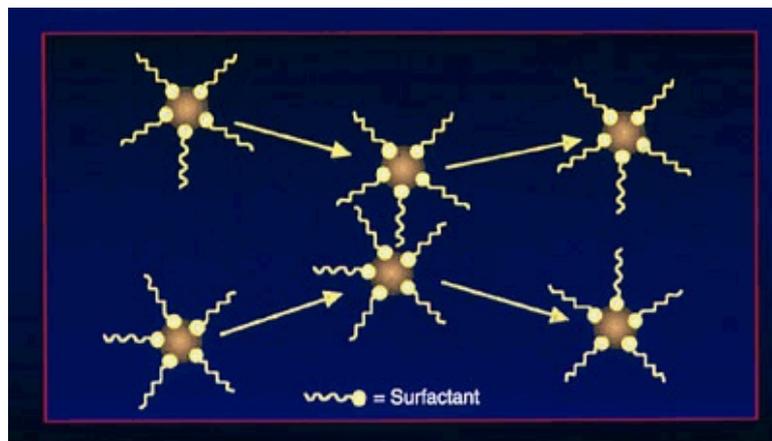
**Figure 3:** Liquid/Liquid Coalescer Medium Construction



**Figure 4:** Coalescing Mechanism/Effect of Surfactant in Free Space



No Surfactant-Coalescing



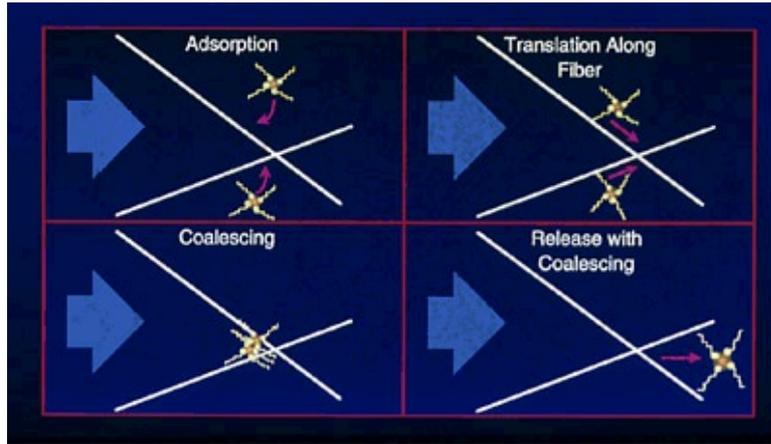
Surfactant-Stable Emulsion

When a fibrous medium is used, the coalescence mechanism can be described by the following steps:

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

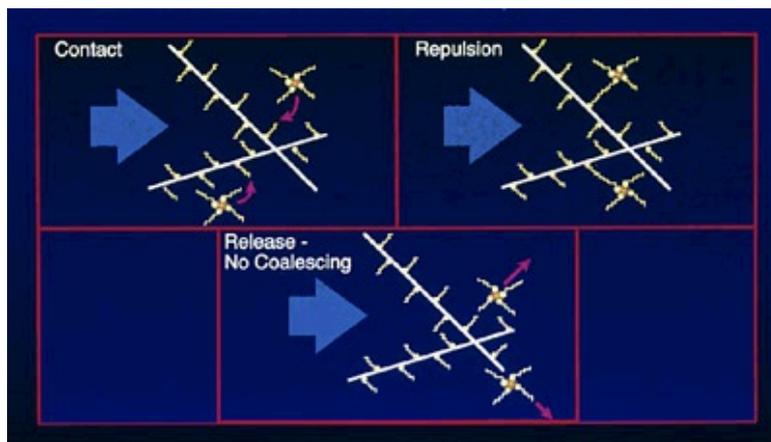
The proposed mechanism is illustrated in Figure 5. Based on this mechanism, we can predict that a number of factors will influence the coalescence performance. The specific surface properties of the coalescer fibers are critical in influencing the adsorption of droplets as well as the ultimate release after coalescing. There is a balancing act between increasing the attraction or adsorption characteristics of the fibers against the release mechanism which strong adsorption would inhibit. The available area for adsorption is controlled by the diameter of these fibers and the length of each of the different porosity zones. The gradient of increasing porosity contributes as well to the coalescence performance. Other considerations for design include pressure drop, cost, compatibility, and disarming by surfactants.

**Figure 5:** Coalescing Mechanism-On Fiber Medium



The necessary condition that droplet fiber adsorption occur for coalescing has been supported by a number of sources<sup>1,2</sup>, although it is not universally accepted. The disarming phenomenon encountered with glass fiber coalescers<sup>3</sup> lends convincing evidence that fiber adsorption is central to the coalescing mechanism. A proposed disarming mechanism of glass fiber coalescers is presented in Figure 6. The surfactant molecules adsorb preferentially to the active sites on the glass coalescer fibers and orient such that the nonpolar tails are facing outward from the coalescer fibers. The dispersed micellar aqueous droplets also have a layer of surfactants in similar orientation with nonpolar tails facing outward. Steric repulsion between the adsorbed surfactants on the droplets and the glass coalescer fibers then interferes with the adsorption of the water droplets and the coalescence is inhibited or disarmed.

**Figure 6:** Coalescer Disarming Mechanism



The presence of significant levels or types of surfactants to cause the disarming phenomenon has been detected by measuring the interfacial tension between the aqueous and hydrocarbon phases. When surfactants are added to the water in a hydrocarbon system, the interfacial tension is decreased and in most cases, an interfacial tension of less than 20 dyne/cm was found to cause disarming of glass fiber coalescers. When specially formulated polymeric coalescer medium was used instead of glass fiber, disarming was not observed. The coalescing performance of a polymeric medium can be greatly enhanced by modification of surface properties that can not be accomplished by glass fiber medium. The explanation for this resistance to disarming is that there is less of an attraction for the polar heads of the surfactants to adsorb onto the coalescer medium fibers. Due to the enhanced surface properties of the new coalescer medium, the surfactants actually adsorb by the nonpolar tails and the polar head are oriented outward where they serve to attract passing water droplets. The coalescer medium thus retains the open sites needed for water droplets to adhere and promote the normal coalescing mechanism.

## Separation of Coalesced Droplets

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Once the droplets have been coalesced they are now assumed to be as large as possible for the given flow conditions. Estimates for the coalesced droplet size can be made using the following equation<sup>4,5,6</sup>

$$d = (k \sigma / \Delta \rho)^n \quad (1)$$

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

Various values for the constant “ $k$ ” have been postulated which are based on a critical Weber number while the value of the exponent constant “ $n$ ” has been reported widely as 1/2 and 3/5. Many of the studies on stable droplet sizes have been conducted at high interfacial tensions ( $> 20$  dyne/cm) and it has been the authors’ experience through actual coalescing tests that low interfacial systems ( $< 20$  dyne/cm) have demonstrated deviations from the Hu and Kintner equation by as much as 100%.

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 and is given by:

$$We = \rho d V^2 / \sigma \quad (2)$$

where  $V$  is the velocity of the droplet. The critical value of the Weber number in liquid/liquid systems has been reported<sup>6,7</sup> between 1-2. Above this critical Weber number the droplets become unstable and break up into smaller fragments. The Weber number increases with droplet diameter, velocity, and decreasing interfacial tension.

The separation stage can be achieved by either a cartridge element with barrier capabilities or by a settling zone which relies on the difference in densities between the coalesced droplets and the bulk fluid.

When the separator cartridge is used, the new system is configured vertically with a patented stacked coalescer and separator. The coalesced droplets leave the coalescer cartridge stage and are repelled by a hydrophobic separator barrier cartridge. The ability of the coalesced droplets to survive the collision with the separator stage is dependent on the Weber number. The ability of the separator to continue to function as a hydrophobic barrier is affected by the surfactants present in a hydrocarbon/aqueous system. Experience has shown that at interfacial tensions below 3 dyne/cm, the separator cartridge becomes ineffective. This may be caused by the inherent fragile nature of the low interfacial tension droplets coalesced or by adsorption of surfactants onto the separator medium that cause a decrease in the hydrophobic nature of the separator medium.

The separation of coalesced droplets is accomplished using a settling zone for bulk hydrocarbon systems when the interfacial tension is below 3 dyne/cm. Here, the coalesced aqueous droplets settle downward by gravity and are collected at a sump at the bottom of the vessel. The purified continuous hydrocarbon phase exits at the top of the housing on the opposite side of the inlet stream.

## Molecular Forces Controlling Coalescence

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The appropriate application of high performance coalescer technology is to separate difficult emulsions. The stability of an emulsion can be measured in terms of interfacial tension. When the interfacial tension is less than 20 dyne/cm, coalescers made of glass fibers can lose separation efficiency. Stable emulsions generally require the presence of at least three components to form. These consist of the two bulk immiscible phases and a small concentration of surfactant. The source of surfactants are numerous in hydrocarbon processing processes. Some of the common sources of surfactants include:

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

The presence of the surfactant leads to the formation of micelles (oil in water) or reverse micelles (water in oil) depending on the relative concentrations of the aqueous and nonpolar phases. The terminology of oil and water are meant to represent nonpolar hydrocarbon and aqueous phases respectively. The formation of micelles in water systems is attributed to London Van der Waal forces between the similar hydrocarbon chains while reverse micelles formed in the oil phase are attributed to hydrogen bonding between the water molecules.

A schematic of a reverse micelle<sup>8</sup> is presented in Figure 7. Evaluation of the stability of emulsions is a complex subject and only a cursory treatment will be given here. Thermodynamics will define the stable equilibrium conditions attainable for a given emulsion system. The key driving forces are the minimization of the internal energy (E) while maximizing entropy (S). For a system at constant volume and temperature the result is that Helmholtz Free Energy (F) is minimized at equilibrium:

$$F = E - TS \quad (3)$$

Interfacial tension ( $\sigma$ ) is defined as the surface energy per area and can be expressed in equivalent units of force per distance (usually dyne/cm). Interfacial tension can also be described as the work required to create additional surface area. The internal energy of the emulsion systems includes a term for the phase boundary such that the energy of the system increases proportionally with increasing area and interfacial tension.

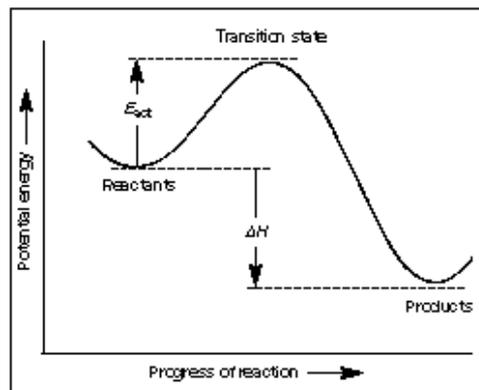
$$E \propto \sigma A \quad (4)$$

where A is the surface area of the droplets and  $\propto$  = [proportional to]

The spherical shape of the droplets itself is the result of minimizing the surface energy per volume. The transition from many small emulsified droplets (high system surface area) to fewer, larger coalesced droplets (low system surface area) results in a net decrease in internal energy for the entire system and hence provides a driving force towards coalescing. There is also a fundamental thermodynamic rationale for decreasing surface tension (lower energy per surface area) to allow emulsions to form with smaller droplet sizes (larger system surface area). The entropy of the system is a measure of disorder which increases with the possible number of configurations. At the limit of minimum entropy, there are two completely separated phases and only one possible configuration exists. As emulsified droplets are formed, many possible configurations are realized and the entropy of the system increases. Thus the tendency to maximize the entropy is countered by the tendency to minimize the internal energy.

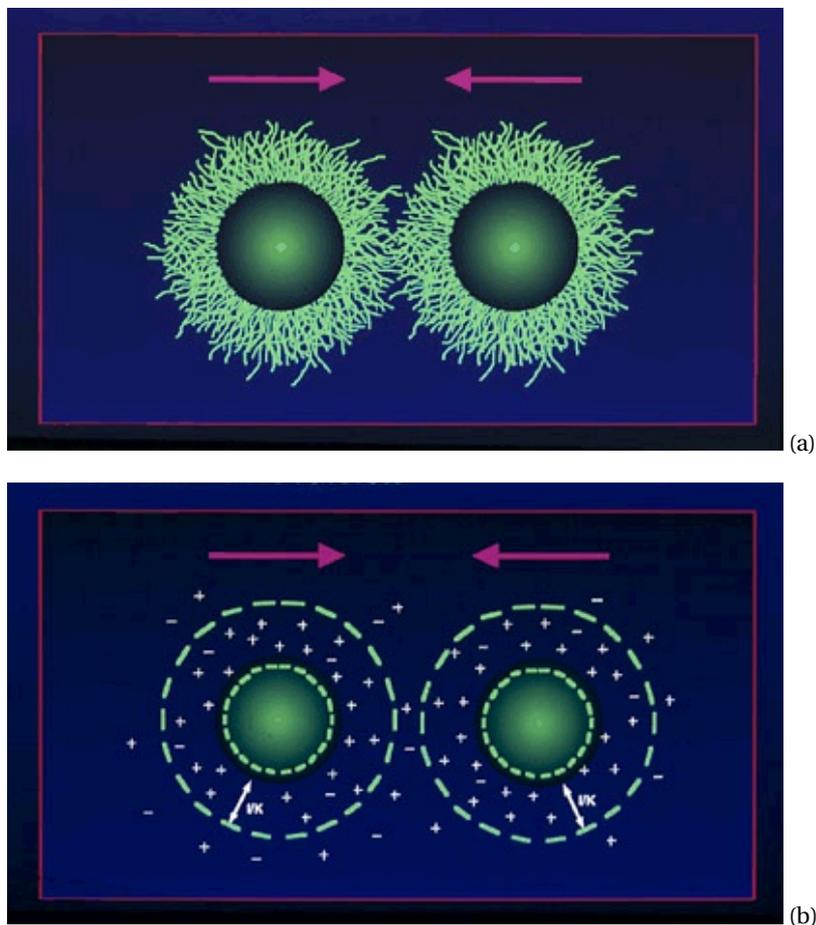
While thermodynamics is useful for predicting the possible equilibrium conditions, most emulsion systems are actually not in equilibrium, but are either moving (slowly) towards the most stable equilibrium state or are fixed in a metastable condition far from the true equilibrium. Kinetics then play a crucial role in determining the state of the emulsion. An analogy with chemical reactions can be made where an energy barrier exists for reactants (uncoalesced droplets) which has to be overcome before the system can move towards the final products (coalesced droplets). A schematic<sup>9</sup> illustrating this concept is presented in Figure 8. The coalescer medium

**Figure 8:** Activation Energy Barrier Model for Coalescing



acts as a catalyst in bringing together the small droplets and overcoming the “activation energy” for coalescence. This process involves passing through a transition state and the energy between the reactants and this transition state is defined as  $E_{act}$  while the difference in the energy state of the reactants and final products is defined as  $\Delta H$ . The energy barriers to coalescing can be classified into steric (spatial) repulsions and electrostatic charge repulsions as are displayed in Figure 9 for the reverse micelle case<sup>10</sup>.

**Figure 9:** Schematic of Steric Repulsion (a) and Electrostatic Charge Repulsion (b) Leading to Stabilization in Reverse Micelles



[Top](#)

### Steric Repulsion

The structure of reverse micelles consist of an inner aqueous phase which is 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 are pointed radially outward. The nonpolar chain sections then sterically repel other micelle formations when they approach at too close of a distance. A number of studies have been conducted in the field of polymer science<sup>11, 12, 13</sup> characterizing the steric repulsion energies of polymer brushes using variations of the Flory-Huggins liquid lattice and excluded volume principle.

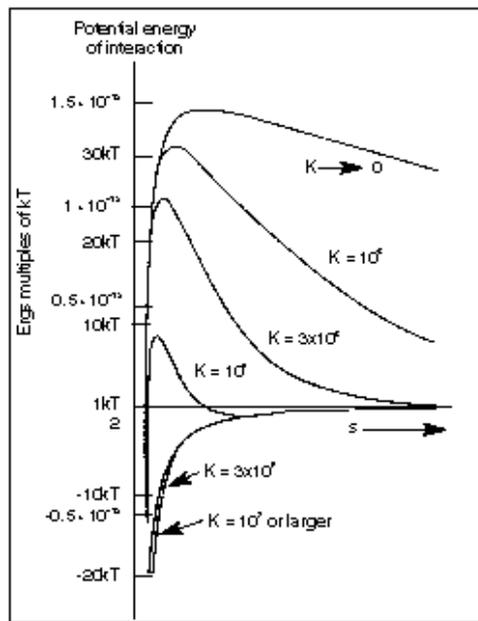
[Top](#)

### Electrostatic Charge Repulsions

The presence of charged species including water, dissolved ions, and charged particulates at the center of micelles gives rise to a balancing electrostatic charge which reaches out a small distance into the continuous phase to preserve electroneutrality. The opposing charge distributions constitute what has been termed the electrical double layer which has been widely studied in the field of electrochemistry with major contributions being attributed to Gouy, Chapman, Stern, and Helmholtz. The charge repulsions between neighboring micelles/reverse micelles occur when the outer electrical double layers which are of the same charge overlap and are repelled.

In the field of colloidal science, modeling of electrostatic repulsion and Van der Waal attractive forces has led to the formation of the DLVO theory (Derjaguin, Landau, Verwey, Overbeek). Some results of DLVO modeling of oil micelles in aqueous solutions are presented in Figure 10 where interaction potentials between spherical particles at different ionic strengths are plotted versus separation distances<sup>14</sup>. As the continuous phase conductivity decreases, the electrostatic repulsion increases considerably deterring aggregation. While most studies have been conducted in aqueous based systems, aggregation of colloidal alumina in nonpolar cyclohexane has led to the conclusion that electrostatic forces are also important in non aqueous based systems<sup>10</sup>. Analogous behavior is expected for reverse micelles in low conducting nonpolar hydrocarbons.

**Figure 10:** DLVO Interaction Potentials Between Charged Spheres in Liquid Media of Varying Conductivity



[Top](#)

## Film Stabilization

The presence of fine particulate can also have the effect of stabilizing the micellar film. Fine particulate such as iron oxides or iron sulfides will accumulate at the interface, stabilizing the micellar boundary. This inhibits the film thinning necessary for two droplets to coalesce. The solid particulate must now be displaced from the film surface before coalescing can occur. Removing the solids with a filter can eliminate some of this added resistance and improve the coalescing efficiency.

[Top](#)

## Overcoming Energy Barriers - Coalescer Mechanisms

The forces which control the adsorption of micelle or reverse micelle droplets onto coalescer fibers may be either electrostatic or London Van der Waal forces depending on the particular material used in the fiber and the system considered. Electrokinetic or Zeta potentials can be measured from streaming potentials caused by flow through fiber beds. In this case, a potential is induced on the fixed fibers due to the flow (shear) of charged species past an electrical double layer<sup>15</sup>. For nonpolar hydrocarbon systems, the low conductivity can offset the low number of charge carriers creating static discharges at high velocities. This phenomenon has led to the creation of anti-static jet fuel additives and velocity restrictions on fueling operations<sup>16</sup>. The sign and magnitude of the zeta potential has been found to vary with fiber materials, fluid composition and temperature. Enhancement of the capture efficiency of Nylon 66 filter membranes for colloidal styrene spheres was found for definite ranges of zeta potential<sup>17</sup>. The filter membrane's removal performance was also found to decrease with the addition of surfactant. This suggests that steric repulsion was now partially countering the electrostatic charging.

[Top](#)

## Commercial Application of Liquid/Liquid Coalescers

New high efficiency liquid/liquid coalescing and separation technology that does not contain glass coalescing media has been tested and are now commercially operating in some gas processing plants and refineries. A discussion of test results and operating results from the following processes is presented below:

### Caustic Treating of Mixed Butanes and Natural Gasoline Process Description

Caustic treating is used to either remove mercaptans, 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 allows for separation of the product from the caustic, which is recirculated to the front end of the treating unit. Problems occur when there is poor separation in the settling drum and caustic carries over into the product.

### Application/Experience

A specially formulated polymeric coalescer which is compatible in 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.

**Table 1:** Comparison of Caustic Removal from Hydrocarbon Between Sand Filter and Liquid/Liquid

## Coalescer

	Highest	Lowest	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

Analysis of the sodium hydroxide was performed using a titration method and the results are based on 13 samples taken over a three week period. To date, there are five operating units worldwide in gas plants and refineries using the polymer coalescer technology to remove carried over caustic from hydrocarbon. An analysis of 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 dyne/cm. This is below the recommended limit of glass coalescers. This low interfacial tension forms a very stable emulsion which is difficult to separate. Surface active sulfur compounds in the spent caustic probably contributed to the extremely low interfacial tension.

A L/L coalescer recently installed on a refinery gasoline system resulted in an annual caustic savings of 126,000 lb/year. The process operating parameters are listed in Table 2. Although the projected caustic savings alone results in a payback in less than one year, greater benefits result from the decrease in off-specification "hazy" gasoline and corrosion of downstream piping and tankage.

**Table 2:** Process Parameters for Caustic Recovery by Liquid/ Liquid Coalescer

<b>Fluid:</b>	Gasoline/ caustic
<b>Flow Rate:</b>	20,100 bbd
<b>Interfacial Tension:</b>	12 dyne/cm
<b>Inlet water/caustic:</b>	2250 ppmv (total)
<b>Caustic Strength:</b>	2.3 % by weight
<b>Outlet Water:</b>	110 ppmv (total)
<b>Projected Annual Caustic Savings:</b>	126,000 lb/year

### Water/Methanol Removal From Propane Process Description

A new process called IFPEXOL-1† employs methanol to permit cooling of the natural gas without causing formation of hydrates or ice crystals. In effect, the methanol acts as an antifreeze. The heart of the Ifpexol process is a special contactor that a) adds the methanol, and b) because of solubility limits on the fluids, extracts water, helping to dry the gas. The gas then passes into conventional refrigeration technology to cool the gas and condense the NGL. This is followed by a three phase separator, that removes NGL for sale, and removes the methanol for reinjection into the Ifpexol contactor.

† Licensed by IFP

### Application/Experience

A L/L coalescer with specially formulated polymeric media was installed on the product NGL stream. Methanol has a high solubility in propane, requiring significant makeup methanol to the loop. To recover and reuse the methanol, a water wash to pull the methanol from the NGL into the water followed by the coalescer was installed to remove the water/methanol solution from the NGL for reinjection into the loop. The excess water was removed in the Ifpexol contactor and the methanol was recycled. After four months of operation with the coalescer and the water wash, methanol losses were reduced by 83% from 120 kg/hr to 20 kg/hr. With methanol prices being both high and volatile, this addition to the process greatly improves the economics resulting in a payback of less than six months.

### Amine Treating of LPG Process Description

Amine treating is used to either remove hydrogen sulfide or carbon dioxide from light liquid hydrocarbons. The reaction takes place in a liquid/liquid contactor tower where the hydrocarbon feed is introduced through a distributor and the amine treating solution is introduced countercurrently at the top of the tower. The sweetened product is removed from the top of the tower. 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/Experience

Field testing has been conducted with a new L/L coalescer under development on this application in three locations. The concentration of the amine downstream of the L/L coalescer was found to be roughly the same as the solubility of the amine in LPG. In one location, the amount of amine coalesced out instantaneously was three times greater than the amount that settled out in a tank with one day residence

time. Carried over amine can be a major contributor to amine losses and the coalesced amine can be recycled back to the contactor. Projected economics for a recently evaluated system at a gas processing plant are summarized in Table 3.

**Table 3:** Projected Economics of Amine Recovery with Liquid / Liquid Coalescer

<b>Flow Rate:</b>	250 gpm
<b>Total Losses:</b>	120 gal/ d
<b>Specific Gravity of LPG:</b>	0.488
<b>Specific Gravity of Amine:</b>	1.05
<b>Concentration of Amine:</b>	40% (wt.) in Water
<b>Total Losses:</b>	333.3 ppm (vol.)
<b>Total Losses:</b>	717.2 ppm (wt.)
<b>Solubility of Amine in LPG:</b>	160.0 ppm (wt.)
<b>Losses Expected From Carryover:</b>	60%
<b>Expected Recovery of Amine:</b>	319.3 ppm (wt.) 148.4 ppm (vol.) 53.4 gal/ d 19,501 gal/yr. 170,773 lbs./yr.
<b>Recovery of total solution:</b>	134 gal/ d 48,753 gal/yr.
<b>Cost of Amine:</b>	\$0.50 lb.
<b>Estimated Savings:</b>	\$85,386 year

In this case, the expected payback again is less than one year. This economic analysis takes into account only recovery of amine losses. When downstream corrosion and equipment fouling and product quality benefits are taken into account, the economics become more attractive. Due to the presence of sulfur compounds, the interfacial tension is again expected to be low. Because LPG flashes when it is brought to atmospheric pressure, an accurate analysis of interfacial tension is difficult.

[Top](#)

## Conclusions

1. Before specifying equipment to separate two liquids, a clear understanding of physical properties including viscosity, specific gravity, presence of surfactants, and interfacial tension is essential.
2. Surfactants lower the interfacial tension of a liquid/liquid mixture and are present in many hydrocarbon processes.
3. The size of a coalesced droplet is a function of interfacial tension, the difference in specific gravities of the two liquids, and the Weber number.
4. Surfactants lower the separation efficiency of glass fiber and a mechanism explaining disarming has been proposed.
5. Energy barriers to coalescing can be classified into steric (spatial) repulsions and electrostatic charge repulsions.
6. New coalescing technology using media not made of glass has successfully separated liquids that contain surfactants and have low interfacial tension.

[Top](#)

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[Top](#)