BIODIESEL MAGAZINE
The World of Biodiesel at Your Fingertips

From the August 2009 Issue

Coalescing Technology for Liquid/Liquid Separations

Liquid/liquid separations, along with solids removal, are crucial to the success of the biodiesel production process. The efficiency of separation significantly influences the quality and overall economics of production.

by Maria Anez-Lingerfelt

When making biodiesel, inefficient separation of glycerol and water can lead to off-spec product, increasing production costs and delays. The choice of separation equipment is influenced by factors such as quality needed, flow rate and solids contamination. Economics also plays a significant role considering initial capital costs, operating costs, waste disposal costs and maintenance. It's important to conduct a thorough process review to fully understand which type of separation technology to use, and the best location for it.

Traditional and present separation technologies used in biodiesel production include gravitational and centrifugal equipment, along with low-efficiency coalescers. Pall's trademarked PhaseSep AS coalescers were developed for liquid/liquid separations in biodiesel processing, and require low energy and cost inputs, and are highly efficient.

Pall's liquid/liquid coalescing technology consists of solids filtration, coalescence and separation (Figure 1). The first step is to remove the solid contaminants using a cartridge filter.

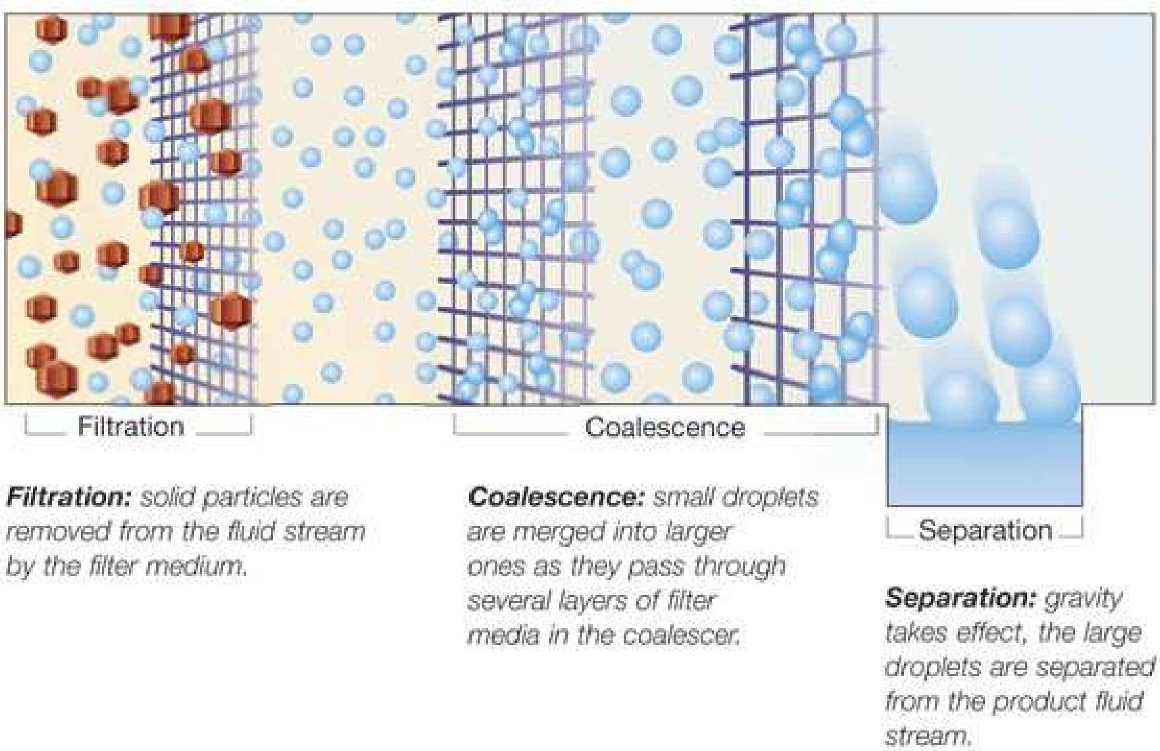

Figure 1. Pall's Liquid/Liquid Coalescing Technology

Solids can increase the stability of an emulsion and can plug the coalescer, thereby reducing its efficiency. Removing solids will precondition the fluid for optimum coalescer performance. During coalescence, the droplets to be separated from the bulk fluid are captured by the coalescer medium. The droplets then move through the coalescer media (with progressively larger pores) and are coalesced to form larger droplets. Lastly, the large droplets are released. After the droplets are released from the media, the phases are separated by gravity in the settling zone.

Pall's PhaseSep AS liquid/liquid coalescer system consists of a coalescer element in a horizontal configuration followed by a settling zone for separation (Figure 2). A prefilter cartridge is placed upstream of the coalescer to remove the solid contaminants. The flow is radial from inside to outside. After settling, the lighter phase exits the top and the heavier phase exits the bottom.

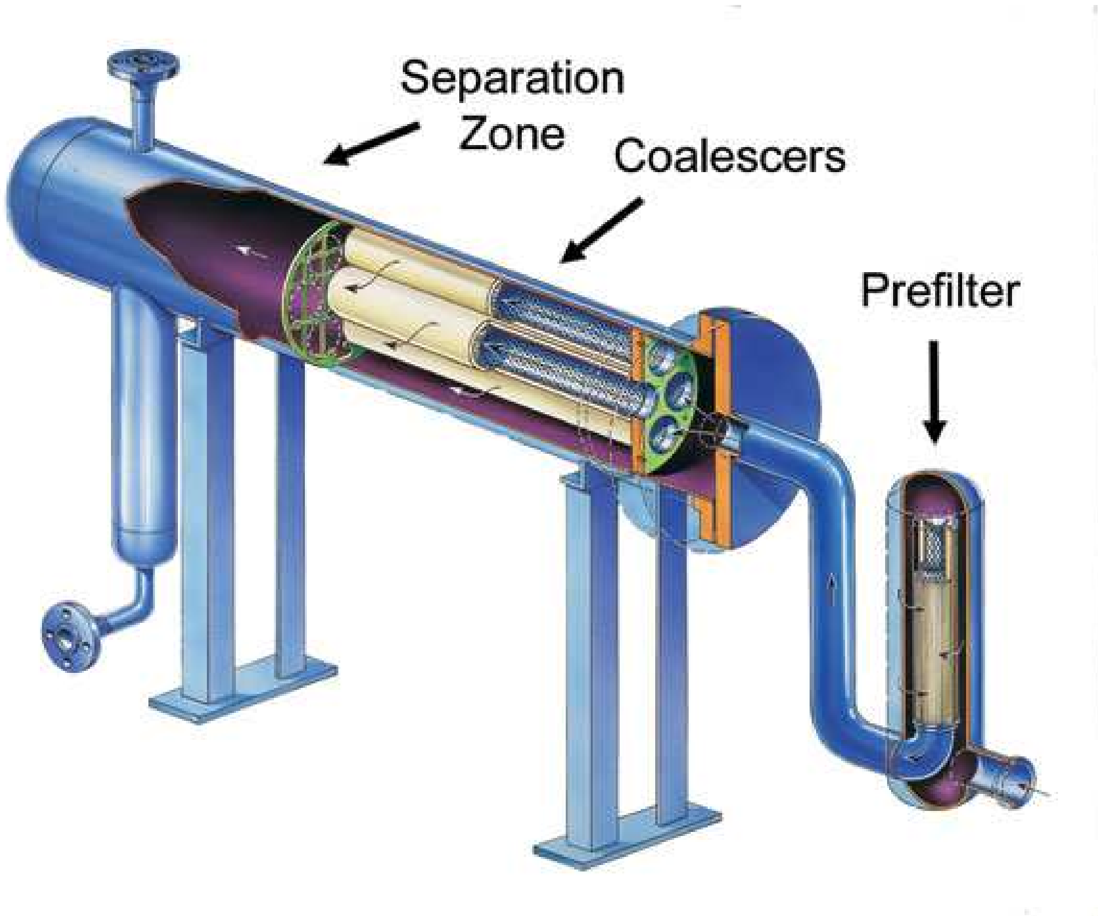

Figure 2. Pall's PhaseSep® AS liquid/liquid coalescer system

Important Factors in Liquid/Liquid Separation
Interfacial tension (IFT), viscosity, density and temperature are four important factors in liquid/liquid separation. Interfacial tension is a measure of the attraction force among each phase for its own species in units of dynes per centimeter (dynes/cm). If the IFT between two liquid phases is high (>20 dynes/cm), they can be separated easily. If the IFT is low (<20 dynes/cm), the separation is more difficult. Pall coalescers are able to capture very fine droplets and break difficult emulsions with IFT as low as 0.5 dynes/cm. Other separation media typically lose efficiencies when the IFT drops below 20 dynes/cm.

Viscosity, or dynamic viscosity, is a measure of a fluid's resistance to flow. Typical units are centipoise (cp) or megapascals (mPa). If the viscosity of one or more fluids in a
mixture is high, it will require greater force or more time to separate them.

Density is the mass per unit volume of the fluid. Typical units are grams per milliliter (g/mL). The higher the difference in density between two fluids, the easier they are to separate. Most physical liquid/liquid separation technologies are based on this principle.

Temperature is a measure of the energy of motion in the fluid's particles—the higher the temperature, the greater the energy of motion. The higher the temperature is, the lower the IFT between two liquid phases is, making separation more difficult.

Pall's PhaseSep AS liquid/liquid coalescer system is currently being used commercially for three liquid/liquid separation applications in the biodiesel production process: the separation of free glycerol after reaction; separation of wash water from biodiesel after water washing; and separation of free fatty acids (FFA), fatty acid methyl esters (FAME) and organic matter-of-non-glycerin (MONG) from glycerin (Figure 3).

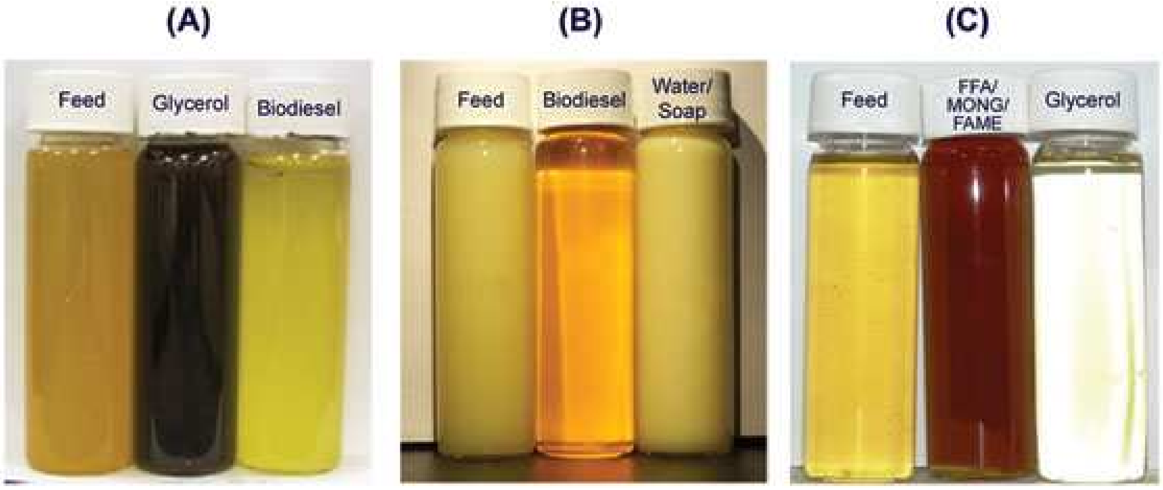

Figure 3. (A) Separation of Glycerol from Biodiesel after Transesterification with methanol present (B) Separation of Water & Soap after Water Wash (C) Separation of FFA/MONG/FAME resulting in water white glycerol product

Separation of Free Glycerol
The feedstock oil is commonly pretreated to remove contaminants that can include phospholipids, FFAs, waxes, sterols, and water among others1, 2.

After pretreatment, the oil is ready to be converted to biodiesel by transesterification, where the oil is mixed with methanol and the catalyst. Important process variables include alcohol-to-oil ratio, choice of catalyst and concentration, and optimum reaction temperature and pressure. Excess methanol drives the equilibrium of the reaction, but using too much methanol is costly and makes the product purification more difficult since methanol acts as a co-solvent between methyl ester and glycerol.

Phase separation is critical to the process. A highly efficient separation of glycerol from methyl ester can lower volumes of water wash or adsorbent/resins (in dry washes) used for downstream purification of the final biodiesel product. Inefficient separation of glycerol can result in lower quality biodiesel and cause injector coking, filter plugging, sediment formation, and can shorten shelf life3. Two common ways to separate the glycerol phase is by draining the bottom layer gravitationally or through settling, or by feeding the mixture into a centrifuge. Settling is time-consuming and prone to product loss at the interface, takes up a large area at the plant and is inefficient. Centrifuges are high in capital and maintenance costs, consume too much energy, are inefficient during variable conditions, and carry over product with the glycerol.

Pall's PhaseSep AS liquid/liquid coalescer system provides the biodiesel industry with an improved option that can achieve high-efficiency separation without long residence times using little energy, and requiring low capital, operating and maintenance costs.

An important factor affecting the efficiency of separation of glycerol is the presence of excess methanol since methanol is a co-solvent between glycerol and biodiesel, resulting in a lower IFT. When the IFT is reduced, the liquids are more difficult to separate and traces of glycerol will remain in the biodiesel phase—and viceversa. Therefore, separation of glycerol before methanol recovery is considered an initial separation step, and additional separation after the methanol is recovered should be considered. Higher efficiency is achieved when a coalescer is placed after methanol removal or if no excess methanol is used. When a two-stage methanol recovery is used, placing the coalescer between the two stages where methanol content may be about 1 to 2 percent has yielded optimum results.

Separation of Wash Water from Biodiesel
Once the glycerol is separated from the biodiesel a water wash may be used to remove any remaining water soluble components. Soap is typically present in the water phase formed by the action of oil and water in the presence of a base (catalyst). In addition, once the methanol has been removed, the remaining free glycerin becomes unsolved and will also be present in the water phase after washing. The ASTM D6751 (and EN 14214) specification for water in final product biodiesel is 500 ppm. Since the solubility of water in biodiesel is approximately 1500 ppm4, the biodiesel is further dried to remove the entrained water.

Efficient separation of the water phase after the water wash will reduce energy inputs to the drier and will also reduce contaminants coming from the aqueous phase. A centrifuge is traditionally used to separate the phases. Pall's PhaseSep AS liquid/liquid coalescer can be placed after the water wash, replacing the centrifuge, to provide a more efficient separation of the aqueous phase resulting in lower free water and reduced contaminants from the water. Use of the coalescer can significantly reduce energy consumption associated with drying.

Separation of FFA, FAME and MONG
It is becoming more important for biodiesel producers to increase revenues by selling the glycerol byproduct. For greater returns, the crude glycerol formed and separated after transesterification must be purified before it is sold.

The main contaminants present in the glycerol phase are free fatty acids, fatty acid methyl ester,(the biodiesel itself), organic matter of non-glycerin, methanol and catalyst. The methanol is recovered and the catalyst is neutralized with an acid. This releases FFA and creates a three-phase system consisting of salts, glycerol and FFA/FAME/MONG. Salt precipitates are removed as solids leaving the glycerol and organic phase to be separated.

Efficient separation of the organic phase (FFA/FAME/MONG) will reduce inputs to the downstream purification steps. Resins and carbon beds will have an extended on-stream life and there will be less fouling in distillation steps. A centrifuge is also traditionally used here. However, Pall's PhaseSep AS liquid/liquid coalescer can be used to provide a more efficient separation of the organic phase, often producing water white crude glycerin and resulting in an improved efficiency for the downstream purification steps.

Conclusion

Biodiesel production continues to increase globally as the need for renewable energy increases. Separations are essential to the success of biodiesel production. For both biodiesel and glycerol products, high efficiency separations upstream lead to a more economical purification downstream. Traditional technologies used in biodiesel production include gravitational equipment, centrifugal equipment, and low efficiency coalescers, all with highly variable results.

Pall's PhaseSep AS liquid/liquid coalescers were developed for biodiesel production separations, require little energy, are low cost and are highly efficient. Pall will continue developing solutions for this highly evolving market to help make biodiesel more economical and sustainable.

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