BIODIESEL MAGAZINE The World of Biodiesel at Your Fingertips

From the November 2006 Issue

Reflux and Recovery

Recycling excess methanol used in the transesterification reaction is a vital function for almost all biodiesel producers. Increasingly, dated distillation techniques are being replaced by more advanced systems.

by Nicholas Zeman

When it comes to the stuff of transesterification, methanol is widely considered the most cost-effective, readily available chemical compound for the job. However, that doesn't change the fact that this well-known alcohol is a volatile, flammable liquid. When handled wrong—or left in too high a concentration in biodiesel—this liquid can be anything from problematic to outright dangerous. That's why specifications are in place to limit its volume to an exact percentage in biodiesel. If that limit is exceeded even slightly, the renewable fuel is deemed off-spec and is technically not salable.

The specification protocol used to keep methanol in check—the ASTM D 92 Pensky-Marten closed-cup flashpoint test—requires that biodiesel not ignite at 130 degrees Celsius (266 degrees Fahrenheit) or lower. The flashpoint of a chemical is the lowest temperature at which a flame will propagate through the vapor of a combustible material to its liquid surface. In other words, it's the minimum temperature at which the liquid produces a sufficient concentration of vapor above it to form an ignitable mixture with air. Note, however, that the ignition source doesn't have to be an open flame. Heat alone will do the trick.

With these facts in mind, it's not difficult to understand why the process of recovering methanol is critical to producing high-quality biodiesel. In a typical biodiesel plant, there are three main components of a methanol recovery system: a still, a distillation column and a condenser. "If your distillation system is improperly designed, you won't get enough methanol out," says Rocky Costello, head of the Redondo Beach, Calif.-based engineering firm R.C. Costello and Associates. "If you don't get enough methanol out, you won't meet the flashpoint test."

In Costello's estimation, each biodiesel plant disaster in the past year—there have been a few reported—has been related to methanol. While not all methanol-related accidents are related to methanol recovery, the process of recouping the chemical can be hairy.

In a conventional biodiesel process, methanol is recovered through assorted distillation techniques (see "Some Distillation Basics" on page 60). Experts say safety is a guiding principle in biodiesel distillation system design and operation. "You're dealing with a very flammable substance, and you're heating it up," says George Hawranika, a process engineer at SRS Engineering in Murrietta, Calif., a designer of distillation systems. "We make sure we have multiple levels of safety and watchdog provisions in our program that monitor multiple sequences. If something goes out of balance, we shut the plant down and inform the operator."

There are essentially two pathways for methanol recovery after the unfinished biofuel exits a reactor where vegetable oils, waste greases or animal fats are transformed into methyl esters: a "wet method" and a "dry method." In the dry method, methanol is distilled straight out of the methyl esters using "ion exchange" technology—a chemical reaction involving the exchange of hydrated ions—or an adsorbent. In the wet method, the methyl esters are "water washed," and the resultant wastewater stream is distilled (in either a batch or continuous process) for methanol recovery.

Continuous Versus Batch

The size of an operation generally influences the selection between batch and continuous distillation options. In large facilities, economics favor continuous systems. "I think the largest batch distillation system I have ever seen is probably about 20,000 gallons," Costello says.

Continuous processes run 24 hours a day, seven days a week (not including scheduled and unplanned shutdowns), unremittingly feeding raw biodiesel in through the side of a fractionation column, taking methanol off the top and leaving marketable fuel on the bottom. "Obviously, the more you run [a facility], and the fewer problems you have, the more money you make because you have more production," Costello says.

Batch systems are usually less capital-intensive than continuous systems but require round-the-clock human operation. On the other hand, continuous systems are generally not amenable to frequent starts and stops. Doing so can cause "unsteady state" conditions that can result in off-spec product. On start-up, a continuous column may not produce ASTM-quality biodiesel until the column reaches a steady state. In other words, all column variables including temperatures, pressures, liquid levels and flow rates should remain constant to produce high-quality fuel.

In a batch-type operation, there is much more operator intervention required, Hawranika says. "On a continuous basis, on the other hand, we have a complete [programmable logic controller] control, where we regulate the entire plant function and keep it within a certain spec much better than [operators can achieve manually]. Very little labor is required on a continuous system because the results are far more accurate."

Batch distillation, however, is very forgiving. This can have its own advantages, Costello says. "If the methanol content in the still is too high at the end of a batch, it is simply run through the still again to remove the residual methanol." Similarly, it is possible for a continuous process to temporarily make product with too much methanol in it. However, once the problem is detected via flashpoint testing, the product would be rerouted back to the distillation column.

Hotter or Higher?

Inside a distillation column, or tower, "reflux" liquid continuously provides cooling and condensation of up-flowing vapors. This action heightens efficiency. Reflux liquid is the portion of the condensed overhead liquid from a distillation tower. Maintaining reflux takes energy in the form of heat. Steam is the preferred mode of heat delivery for this process because it is widely considered safer and more efficient than electrical heat. If electrical heat is used, it requires explosion-proofing. "On large systems, steam is our main choice, but we can use hot oil systems or electrical systems, as well," Hawranika says. "When you get into larger systems, the electrical energy requirement gets way too high for most plant applications."

The more reflux provided, the more effectively a distillation column can separate materials with different boiling temperatures. High reflux levels require high heat, however, and high heat requires high energy costs. There's a caveat next to that rule, though. Costello says producers are able to use less reflux—and subsequently less heat—by using a taller distillation column for the same degree of separation. "There's always a trade-off of operating costs versus installed costs," Costello says. "A taller, more expensive column has lower energy cost, and a shorter, less expensive column has higher energy costs."

Hawranika says the company's design principles allow for the recovery of nearly 30 percent more methanol than some other systems. "There are companies that are expecting only a 65 percent to 70 percent methanol recovery from that biodiesel stream," he says. "That's good,but if you can get 99.5 percent out, that's twice as good. That's one advantage of what we're offering, and yes, there's a lot more design [that goes] into it. There's a lot more money that's invested on our end to conduct chemical simulations [that] provide us with the facts … and we build to that."

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