ABSTRACT

DISTILLED ALCOHOLIC BEVERAGE PRODUCTION USING REACTIVE DISTILLATION TECHNIQUES

By

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The goal of this research was to couple industrial beverage distillation with a chemical reaction to create a flavored spirit without adding anything after the final distillation. Ethanol reacted with butyric acid in the final distillation over Amberlyst® 15 to create ethyl butyrate, which gave the spirit a fruity smell comparable to Juicy Fruit® gum.

It was shown that if a beverage ethanol fermentation having 63 ± 3 g/L ethanol and 1 g/L butyric acid were distilled, the low wine will have 357 ± 32 g/L ethanol and 1.4 ± 0.1 g/L butyric acid. This sets up the low wine to be distilled over the catalyst to create the ethyl butyrate.

For the study, low wine was obtained from a craft distillery. This gave the most accurate representation of how this system would act in the beverage industry. Butyric acid was added to this low wine and distilled on a glass vigreux column with copper wire on the inside to simulate an industrial copper distillation column. The distillations started with a pot ethanol concentration of 30% ABV and varied: the butyric acid starting concentration from 0.5 g/L to 5 g/L, the catalyst loading from 1 g dry 1 L to 100 g dry/L, and the catalyst position in the column from being in the pot, the bottom of the column, and the top of the column.

All compounds, except butyric acid, followed the ethanol distillate concentration curve and dropped to zero as the ethanol ran out of the system. The butyric acid was not present in the distillate until the ethanol concentration in the distillate started to decreased. As the butyric acid starting concentration increased, the butyric acid in the distillate increased.

The ethyl butyrate concentration was a function of all three variables mentioned above. As
starting butyric acid concentration increased, so did the ethyl butyrate. As the catalyst loading increased, the ethyl butyrate concentration increased. When the catalyst was located in the pot, the ethyl butyrate was shown to distill during the entire distillation only to drop with the ethanol. When the catalyst was in the bottom of the column, ethyl butyrate was present in the beginning but the concentration increased to approximately doubling its starting concentration half way through the distillation. When the catalyst was in the top of the column, the ethyl butyrate was severely delayed in the distillate, only to distill through the tails section and again fall with the ethanol.

During distillation, lower boiling components vaporize and go up the column, making the bottom of the column a higher temperature than the top. Thus, there is more water and more butyric acid at the bottom of the column where the boiling point of the liquid is higher. As ethanol was depleted, the higher temperature water front rose past the catalyst and ultimately to the top of the column where it came out as distillate, bringing butyric acid with it. When this front reached the catalyst, the butyric acid reacted with ethanol to create the ethyl butyrate, which was carried up in the vapor phase by the ethanol and out in the distillate. This unsteady-state system explains why ethyl butyrate presence in the distillate was largely controlled by the location of the catalyst, and butyric acid was present only after ethanol had been depleted.

Chemical equilibrium for this system of esterification and catalyst has been shown to take 300 minutes. The reactive distillation system presented has at most 1-2 minutes of catalyst contact time. Thus, the reaction system is not in chemical equilibrium, but rather is kinetically controlled by the distillation.

Assuming a Murphree efficiency of 75%, it was determined that this system has 2.4 equilibrium stages.

A provisional patent application has been filed for this process.