Increasing refinery biofuels production

The benefits of producing bio-ethers with catalytic distillation compared to blending bio-ethanol are addressed. Options such as the conversion of MTBE units to ETBE production, combined with skeletal isomerisation, can provide higher volumes of high-octane gasoline

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The European Union has set challenging targets for European refiners by increasing the minimum biofuel content in fuels to 5.75% by 2010. As a result, refiners are investing in process units to produce biofuels such as bio-ETBE (ethyl tertiary butyl ether), which is produced by the etherification of bio-ethanol and isobutylene.

In general, the investment cost to produce bio-ETBE is very low because existing MTBE units can be revamped to produce ETBE. The use of bio-ETBE is preferred, because the blending of bio-ethanol into gasoline can lead to water separation and an unwanted increase in gasoline vapour pressure. Bio-ETBE is now a stable and reliable biofuel component.

To meet a 5.75% biofuel content by 2010, the contribution of bio-ETBE will be too small, even if all available isobutenes are converted to bio-ETBE. Refiners recognise the advantage of using bio-ethers as a stable biofuel component, so other technologies are required to maximise the production of bio-ethers. The first logical step is to utilise the same time-proven process for the etherification of C5 iso-olefins, which is a very cost-effective option for bio-ether production, since C5 iso-olefins are available in sufficient quantities in most refineries. The added advantage is that C5 components that have a high vapour pressure are converted to higher-octane bio-ethers with a low vapour pressure.

The second step is to use skeletal isomerisation technology to convert normal butenes and pentenes to reactive iso-olefins. With this technology, iso-olefin production is increased by 50-100%, which results in a similar increase in bio-ether production.

Refiners who have previously invested in the production of bio-ethers are aware of their benefits and are now considering a further increase in production, either through the production of bio-TAEE or through skeletal isomerisation, or a combination of both.

For gasoline blending, European refiners have a choice of blending bio-ethanol or bio-ethers produced from bio-ethanol and refinery iso-olefins. Several European refiners have already opted to produce bio-ethers because it offers significant advantages over the blending of ethanol.

EU directive

The EU maintains broad objectives towards improving the security of energy supply, reducing greenhouse gas emissions and creating new opportunities for sustainable, rural development. To meet these objectives, it promotes the use of biofuels, which would replace diesel or gasoline for transport purposes. Directive 2003/30/EC has been established to oblige EU member states to use a certain amount of biofuels, while directive 2003/96/EC provides an opportunity for EU member states to allow an excise duty reduction, thereby promoting the use of biofuels.

The former directive calls for a minimum of 2% biofuels relative to the amount of gasoline and diesel sold in 2005, growing to 5.75% in 2010. The percentages indicated are based on energy content. For bio-ethanol, an energy content of 5.75% biofuels corresponds to approximately 8.5 wt% ethanol in gasoline for ETBE, to approximately 14 wt% ETBE in gasoline.

Excise tax incentives

Under normal circumstances, the cost of bio-ethers production is higher than the price of comparable fossil fuels like gasoline. In most European countries, excise tax is a significant portion of the gasoline price, so governments have the option to promote the use of biofuels via excise tax incentives. Austria, France, Germany, Italy, Spain, Sweden and the UK have implemented the directives efficiently and offer either full tax exemption or partial tax exemption on the use of biofuels.

With the EU directive in place, some countries offered attractive tax incentives, which resulted in initiatives by refiners and others to produce biofuels. Specifically for the production of bio-ethers, several refiners who operated an MTBE unit opted to revamp their units to produce ETBE. Often, the change from MTBE to ETBE required very few modifications to the MTBE unit. Most refiners opted to modify the unit with minimum changes and accept a lower conversion and/or a lower throughput. Regardless, the change from MTBE to ETBE production resulted in a significant amount of bio-ether production.

Since the production of biofuels is not mandated in Europe, refiners still use the flexibility of these etherification units to return to MTBE production, especially when MTBE prices are high. Presently, some 63% of the etherification units in Europe have or are producing bio-ethers. The majority of these are located in Germany, France and Spain.

The cost of revamping an existing MTBE unit to produce ETBE is very low. The majority of bio-ether capacity is produced by units that previously produced MTBE. Another option for the production of bio-ethers is to build a grassroots etherification unit. The investment cost of a grassroots unit is obviously higher than that of a revamped MTBE unit, but still moderate because these units operate at low pressure and have few pieces of
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steam crackers. In addition, iso-catalytic cracking (FCC) units and olefins are available mainly from fluid with bio-ethanol. The reactive iso-olefins (eg, iso-butylene) are produced by the dehydration of tertiary butyl alcohol (TBA), a byproduct from propylene oxide/tertiary butanol manufacturing.

Currently, some 20% of the ether production in Europe is based on isobutylene originating from TBA dehydration, and approximately 30% is based on isobutylene from steam crackers, which leaves 50% for iso-olefins from FCC units (~3000 kMTA). Additional isobutylene is unlikely to come from steam crackers or from TBA dehydration in the near future because very few new steam crackers and propylene oxide/tertiary butanol units are planned. It is therefore expected that growth in iso-olefin capacity will come from FCC units.

The overall FCC unit capacity in Europe is approximately 2500 000 BPSD. If all these used their iso-olefins to produce bio-ethers, bio-ethers production from FCC offgas would increase from 3000–9500 kMTA. The major contributor to this increase (~4500 kMTA) would be the production of heavier bio-ethers such as TAE from C_5 and heavier iso-olefins.

If all FCC iso-olefin capacity was used together with the ether capacity produced from TBA and steam cracker isobutylene, the total amount of bio-ether that could be produced amounts to 13 000 kMTA. In energy terms, this is 10.5 million tons of oil equivalent (Mtoe), or 7.5% of the expected 2010 gasoline energy demand (142.1 Mtoe). Corrected for the bio-ethanol content, this represents 3.3% of the gasoline energy demand.

Of course, this would apply only if all refiners with an FCC unit decided to produce bio-ethers from both C_5 and C_6 iso-olefins. This is unlikely, but it shows that most potential bio-ether production would be from processing heavier iso-olefins.

These numbers are estimated based on processing iso-olefins produced directly from the FCC unit. Bio-ether production could be increased further through skeletal isomerisation of the raffinate from the ETBE and/or TAAE units. With this step, bio-ether production could be increased economically by approximately 60%. As a result, the biofuels content would increase from 3.3–5.3% of the gasoline energy demand.

**Options for biofuels blending in gasoline**

Current European gasoline specifications call for a maximum ethanol content of 5 vol%, a maximum ether content of 15 vol%, a maximum oxygen content of 2.7 wt%, and a maximum olefins content of 18 vol% (with pressure for reduction to 10 vol% by the Worldwide Fuels Charter).

The present constraints for ethanol, ethers, oxygen and biofuels content are represented graphically in Figure 2. The ethanol constraint is shown on the Y-axis at 5 vol%, and the ether constraint of 15 vol% is shown on the X-axis. In a mixture of 5.0 vol% ethanol and approximately 6.0 vol% ether, the oxygen constraint becomes limiting, so less ethanol is allowed. Similarly, in a mixture of 15% ether, approximately 1.0 vol% ethanol can be blended before the oxygen constraint becomes limiting. The line connecting the 5.0 vol% ethanol constraint to the 15 vol% ether constraint shows the constraints for ether, ethanol and oxygen. Also shown in the graph is the biofuels energy content, where the shaded area represents a biofuels energy content of less than 5.75%.

It is clear that with the 5.0 vol% ethanol limitation, the 5.75% energy content specification would not be met. With bio-ethers, the bio-energy content
can be met, while the octane gain is also higher.

Discussions are ongoing on the revision of the fuel quality directive, and specifically on the biofuel content (as bio-ethanol or bio-ether), oxygenate content and Reid Vapour Pressure (RVP) limitations. In a recent parliamentary proposal of 19 July 2007,7 which would amend the present fuel quality directive 98/70/EC, the introduction of a biofuel-grade gasoline is proposed. Its specification allows for higher ethanol, oxygenate and bio-ether content.

The constraints for the biofuel-grade gasoline are represented graphically in Figure 3. Although the energy content of this special grade exceeds the required bio-energy specification of 5.75%, the bio-energy content of the full gasoline pool (ie, normal gasoline and biofuel gasoline) will most likely still be below the requirements. However, when blending bio-ethers, the bio-energy content requirements of the full gasoline pool will be met.

**Volatility**

The present RVP specification, as set by directive 98/70/EC, is a maximum of 60 kPa. In the parliamentary proposal of 19 July 2007, the RVP specification would be lowered from 60–56 kPa. For biofuel-grade gasoline, the intent is to provide a waiver of 4 kPa when 3–10% biofuels is included. The EU recognises that air quality deteriorates at a higher RVP.

The volatility of heavier bio-ethers, such as ETBE or TAAE, is significantly lower than the volatility of ethanol. When blending ethanol, the volatility — in terms of RVP — is a constraint, especially when small amounts (up to 10 vol%) of ethanol are blended. The RVP of ethanol is 124 kPa, whereas the RVP of ETBE and TAAE are 27.5 kPa and 7 kPa respectively. At higher levels of ethanol (ie, >10%), the RVP of the gasoline/ethanol mixture decreases but is still higher than the RVP of gasoline alone.

A reduction in RVP from 60–56 kPa will force refiners to reduce the amount of light components in the gasoline pool. The waiver of 4 kPa that is offered for biofuels in the range of 3–10% will help, but the waiver is insufficient when blending ethanol. As shown in Figure 4a, when blending base gasoline with 5 vol% ethanol, the base gasoline should have a maximum RVP specification of 53 kPa because after blending with ethanol the RVP will increase from 53–60 kPa.

Since the RVP of bio-ethers is significantly lower than that of gasoline, when blending 15 vol% TAAE the RVP will drop. To meet the RVP specification of 60 kPa for a blend containing 5 vol% TAAE, the RVP of the base gasoline could be as high as 67 kPa (Figure 4b). With TAAE blending, the refiner is less likely to run into an RVP constraint because the maximum RVP of the base gasoline with TAAE blending (67 kPa) is significantly higher than that with ethanol blending (53 kPa).

When the maximum amount of bio-ethers has been blended in the gasoline pool, the maximum oxygen specification has not yet been reached, so blending additional ethanol is still possible. For the situation shown in Figure 2 (15% TAAE, ~1.5% ethanol), RVP would be reduced from 63 kPa (base gasoline) to 60 kPa (blended gasoline).

For the biofuels-grade option shown in Figure 3 (22% TAAE, ~1.5% ethanol), the waiver of 4 kPa would not apply because the amount of biofuel blended is more than 10%. Due to the larger amount of TAAE blended (22%), the RVP reduction is higher than the RVP reduction for 15% TAAE. In this case, the RVP would be reduced from 63 kPa (base gasoline) to 56 kPa (blended gasoline).

**Octane**

The octane number of heavy bio-ethers is higher than required for the gasoline pool. Although the octane number of ethanol (RON 129; MON 102) is higher than that of ETBE (RON 118; MON 101) or TAAE (RON 105; MON 95), the fact that more bio-ethers can be blended in the gasoline pool gives a higher octane value. Overall, the octane value of the gasoline pool increases by two RON octane points when blending 5.0 vol% ethanol, while it increases by 4.3 RON octane points when blending 15 vol% ETBE, and 2.3 RON when blending 15 vol% TAAE. This extra octane helps to replace that lost when the gasoline sulphur is reduced to current specification levels.

**Olefins**

Olefins in FCC naphtha contribute significantly to the octane number of
Consideration for bio-ether production in a refinery

Given the potential advantages of blending bio-ethers into the gasoline pool, bio-ether production is currently being considered by several European refiners. The most cost-effective option is to revamp an existing MTBE unit to produce ETBE. The investment cost for such an option depends on the flexibility of the existing equipment and is usually in the order of $20–100/BPSD FCC capacity. Alternatively, the refiner could opt to install a new ETBE unit. The ISBL investment cost is about $150–250/BPSD FCC capacity.

As mentioned earlier, the availability of isobutylene is limited, while C₅ isoolesfins are available in most refineries with an FCC unit. The ISBL investment cost of a grassroots TAEE unit is approximately $200–300/BPSD FCC capacity.

For refiners who want to increase iso-olefin capacity, skeletal isomerisation may be an interesting option. Figure 5 shows the implementation of this technology. The skeletal isomerisation process uses raffinate from the etherification process and effectively converts C₅ and C₆ normal-olefins to iso-olefins. To maximise conversion, the effluent from the skeletal isomerisation unit is recycled back to the etherification unit. However, the raffinate from the etherification unit also contains butanes and pentanes that are not converted. Butanes and pentanes will build up in the recycle and therefore a purge is required to create an outlet for the butanes and pentanes.

The ISBL investment cost of a grassroots, combined TAEE/isomerisation unit is about $500–800/BPSD FCC capacity. The capacity of such a unit is approximately 40–80% higher than that of a TAEE unit alone. Payout times for an investment in grassroots etherification units for the production of biofuels, using CDTech technologies, are generally six months to a year.

Pretreatment

Refinery C₁₀₅ and light catalytic naphtha (LCN) are available as etherification feedstocks. The diolein content of the C₁₀₅ is typically less than 0.3 wt%, while the LCN may contain as much as 2.0% pentadienes and hexadienes. The butadiene is not very reactive at etherification conditions and therefore poses no problem. However, the LCN diolefins are quite reactive and tend to form oligomers at etherification conditions. The oligomers foul the primary reactor etherification catalyst as well as the ether column reboiler; frequent shutdowns are required to replace the catalyst and clean equipment. In addition, the oligomers result in unacceptably high gum levels.

<table>
<thead>
<tr>
<th>bio-ethers</th>
<th>n-C₅=, i-C₅=</th>
<th>n-C₆=</th>
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<tbody>
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<tr>
<td>n-C₆=</td>
<td></td>
<td>Skeletal isomerisation</td>
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</table>

Figure 5 Possible routes for etherification

Figure 6 CDHydro column

the gasoline. However, the FCC naphtha needs to be hydrotreated in order to produce low-sulphur fuels, with the adverse effect that the olefins are also saturated, resulting in a lower octane number.

An interesting option is to separate the light FCC naphtha (pretreated for mercaptan and diene removal) and to subsequently produce heavy bio-ethers such as TAEE through etherification. Overall, the total amount of olefins is reduced significantly, while the octane number is increased through etherification. This also helps to offset the octane loss due to olefin saturation that results from desulphurisation of FCC gasoline.

Using the present biofuels specifications shown in Figure 2 and a 32 vol% olefins content in base gasoline as a starting point, blending 5.0 vol% ethanol will result in an olefins reduction from 32–28 vol%. Using the iso-olefins in the base gasoline to produce TAEE will result in a reduction in olefins content from 32 vol% to less than 18 vol%.

Dilution effect

Since bio-ethers do not contain aromatics, the heavy bio-ethers have a dilution effect on the amount of aromatic hydrocarbons in the gasoline/bio-ether mixture. Assuming the mixture contains 35 vol% aromatics, the total aromatics content drops to 30% when 15% bio-ether is blended into the gasoline pool.

Economic advantage

Depending on the tax incentive (or tax penalty) applicable, biofuels production is interesting from an economic point of view because bio-ethers production allows more biofuels to be blended into the gasoline pool. Since the octane value increases when bio-ethers are blended, the severity of the reforming unit can be reduced, or the refiner does not need to purchase other high-octane components to meet specifications. Additionally, when blending bio-ethers, the olefin content and Rvp are reduced. This offers an economic benefit, especially when these are refinery constraints.

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when the bio-ether product is blended into finished gasoline.

To prevent this problem, the diolefins must be removed from the LCN by selective hydrogenation. Conventional technologies use a fixed-bed catalytic reactor in combination with a distillation column to hydrogenate the diolefins and separate the LCN from the heavy catalytic naphtha. Unfortunately, the oligomers still form in the catalyt bed, resulting in reduced catalyst activity and an increased pressure drop. As a result, it is necessary to periodically shut down the reactor for catalyst replacement, necessitating the use of a spare parallel reactor to continue the diolefin removal and downstream processes.

A catalytic distillation application that addresses these problems has been developed and commercialised. The proprietary CDHydro process combines selective hydrogenation and distillation. It is applied by placing selective hydrogenation catalyst contained in structured distillation packing (CDModule) in the top of a gasoline splitter and then adding hydrogen below the catalyst (Figure 6). The CDHydro catalyst bed operates at pressures significantly lower than conventional fixed-bed reactors. As a result, hydrogen consumption is substantially reduced.

In the bottom of the CDModule zone, mercaptans react with diolefins to form olefinic sulphides (Figure 7). These heavy sulphides have higher boiling points than the light catalytic naphtha (LCN) fraction and are easily fractionated to the bottom product, effectively desulphurising the LCN. Unlike disulphides from caustic sweetening, olefinic sulphides are thermally stable and do not decompose in the reboiler to cause other problems. The overhead stream is desulphurised without the use of caustic, and essentially all sulphur leaves the column with the bottom product. The CDHydro product typically contains less than 1.0 ppm mercaptans and can be blended directly into the gasoline pool.

Additional hydrotreating functions are achieved in the CDHydro column. In the upper section of the catalyt bed, hydrogen reacts with diolefins to selectively produce olefins. The overhead stream is low in diolefins, reducing gasoline gum formation and improving the quality of the LCN for etherification feedstock. Other benefits of selective hydrogenation are reduced Rvp and increased octane of the C5 cut. The double-bond isomerisation accompanying selective hydrogenation is responsible for both effects. Moving the double bond from the α to the β position on the molecule converts 3-methyl butene-1 to 2-methyl butene-2 or 2-methyl butene-1, and converts pentene-1 to pentene-2. In both cases, the β-olefin has lower vapour pressure and higher octane than the α-olefin.

This application of the CDHydro process has been commercialised in more than 20 refineries. The first installation for treating LCN has been in operation for more than ten years and is still performing all the previously described functions using the original catalyst load, with no detectable loss of catalyst activity. The process has demonstrated high reliability and robustness in the refinery environment.

**Etherification**

For the production of MTBE, ETBE, TAME and TAEE, CDTech uses processes (over 80 units worldwide) based on catalytic distillation. They are based on a two-step reactor design, consisting of a boiling-point fixed-bed reactor, followed by final conversion in a catalytic distillation column. The process utilises an acidic ion exchange resin catalyst in both the fixed-bed reactor and the proprietary catalytic distillation structures.

The boiling-point reactor is designed so that liquid is allowed to reach its boiling point by absorbing the heat of reaction, after which a limited amount of vapourisation takes place, thereby maintaining precise temperature control. The maximum temperature is adjusted by setting the total system pressure. Since the reacting liquid mixture temperature cannot exceed the boiling temperature, control is far superior to those systems in which heat must be transferred by convection or conduction. This design retains the heat of reaction as latent heat, reducing heat input requirements for the ensuing fractionation. Reactor effluent is cooled by condensation rather than by convection, resulting in the use of smaller-sized equipment.

The catalytic distillation column, which combines reaction and fractionation in a single unit operation, allows a high conversion of iso-olefins (exceeding fixed-bed limitations) to be achieved simply and economically. By using distillation to separate the product from the reactants, the equilibrium limitation is exceeded and a higher conversion of isobutylene is achieved. Catalytic distillation also takes advantage of improved kinetics through increased temperature without penalising equilibrium conversion.

The equilibrium conversion for the production of several ethers is shown in Table 1. In general, C5 iso-olefins are routed to an etherification unit where very high conversion to ethers is achieved.

Specifically, for C5, and heavier iso-olefins, single-pass conversion is very low and a substantial benefit can be achieved by using catalytic distillation. In the CDTech process, the C5 and heavier iso-olefins are converted in the boiling-point reactor to near-equilibrium conversion. Unconverted C5 and heavier iso-olefins do not reach the catalytic distillation zone in the fractionator and are thus not further converted. While further conversion in a separate distillation column is possible, the addition of a separate distillation column cannot often be justified economically.

Other conventional processes make use of a side-draw from the fractionator (containing unconverted iso-olefins and ethanol) that is recycled back to the etherification reactors. When recycling iso-olefins, the overall conversion of all iso-olefins is higher. However, the additional C5 iso-olefin conversion is relatively low compared to what can be achieved with catalytic distillation.

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**Table 1**

<table>
<thead>
<tr>
<th>Reaction</th>
<th>Equilibrium conversion</th>
<th>CDTech conversion</th>
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<tbody>
<tr>
<td>C5, iso-olefins to ETBE</td>
<td>90</td>
<td>97</td>
</tr>
<tr>
<td>C5, iso-olefins to TAME</td>
<td>50</td>
<td>95</td>
</tr>
<tr>
<td>C5, iso-olefins to TAMEE</td>
<td>25</td>
<td>95</td>
</tr>
<tr>
<td>C5, iso-olefins to THPEE</td>
<td>20</td>
<td>95</td>
</tr>
</tbody>
</table>

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**Figure 7** Mercaptan removal reaction

![Figure 7](image-url)
Overall, the amount of iso-olefins converted using catalytic distillation (with more C$_5$ iso-olefins converted) is similar to the amount of iso-olefins converted using the process with recycle operation (with more C$_4$ and heavier iso-olefins converted). However, the recycle process consumes significantly more utilities due to the large recycle stream, resulting in utilities costs that are two to three times higher. The high recycle rate not only results in higher operating costs, but also in higher investment costs.

**Skeletal isomerisation**

The CDTech IsomPlus technology for the skeletal isomerisation of normal butenes and normal pentenes employs a zeolite-based catalyst. Specifically developed for this service, the catalyst provides near-equilibrium conversion of normal butenes or pentenes to iso-olefins at high selectivity and with long process cycle times. The process configuration is simple, and moderate process conditions result in low capital and operating costs.

Hydrocarbon feed, such as ETBE or TAEE raffinate, is vapourised and superheated prior to entering the skeletal isomerisation reactor. The hydrocarbon feed does not require steam or other diluents, or the addition of catalyst activation agents, to promote the reaction. The vapour stream passes through the fixed-bed reactor where the conversion takes place.

In the C$_4$ IsomPlus process, up to 44% of the normal butenes are converted at greater than 86% selectivity to isobutylene. The reactor effluent is cooled and compressed to a fractionation column, where the C$_4$ fraction is separated from a gasoline fraction and recycled to the ETBE unit.

In the C$_5$ IsomPlus process, up to 66% of the normal pentenes are converted at greater than 95% selectivity to isoamylene. The reactor effluent is condensed and pumped to the etherification unit. A purge is taken from the IsomPlus feed to limit the build-up of saturates in the recycle stream.

During the process cycle, coke gradually builds up on the catalyst, reducing the isomerisation activity. At the end of the process cycle, the feed is switched to a fresh catalyst bed. The spent catalyst bed is regenerated by oxidising the coke with an air/nitrogen mixture.

**Conclusion**

The EU has provided challenging targets to implement biofuels. Several countries either promote or mandate the use of biofuels through tax incentives or tax penalties. Refiners have the ability to blend bioethanol directly. However, blending ethanol results in a significant increase in gasoline vapour pressure, which is a constraint for many refiners. An attractive solution is to react bio-ethanol with refinery iso-olefins to produce bioethers and at the same time reduce the olefins content. Bio-ethers such as ETBE and TAEE are excellent blending components due to their low volatility, high octane number and the fact they do not contain aromatics or olefins.

Many refiners who already operated an MTBE unit have decided to revamp it to produce ETBE. The cost of such a revamp is usually very low and offers a cost-effective way to produce biofuels. Only a few MTBE units have not yet been converted, but are expected to convert to ETBE production in the near future.

The next most cost-effective option is to build a grassroots etherification unit, since these units involve few pieces of equipment, operate at low pressure and do not require a significant investment. The production of bio-ethers from heavier iso-olefins (C$_5$ plus) that are normally present in light FCC naphtha could be of particular interest for refiners because it offers an option to reduce the olefins content, increases the octane number and reduces the volatility.

Etherification processes that make use of time-proven catalytic distillation technology are available. Skeletal isomerisation is also available to increase the production of bio-ethers even further. For the production of bio-ethers, these processes offer excellent economics in terms of investment and operating costs and run length. Payout times for an investment in the production of bio-ethers using CDTech technologies are generally six months to a year.

With the European drive to increase the production of biofuels in the next few years, refiners will also be evaluating options to meet the demand for biofuels.

**References**