Produce trans-free fats

Interchem® Technology

Principles
Interesterification consists in changing the physical properties of the oil by an interchange of the fatty acids in between and within the different composing triglycerides.

Interesterification is used to:
- change the overall melting profile (usually to smoothen the solid fat content) or the melting point of the mixture (usually a decrease)
- improve the compatibility of the different triglycerides in the solid state
- improve the plasticity of the resulting solid by changing the (re)crystallisation properties
- combine the properties of mixed oils and fats

Ester-ester interchange can be achieved without catalyst at high temperatures (above 200°C).

In the presence of an acid, alkaline or metallic, the reaction may however take place under much milder conditions (20- >100°C).

The most effective catalysts are the alkali (m)ethylates and Sodium/Potassium alloys.

Interesterification is a 2 – stage process. The metal catalyst normally acts as “pre-catalyst”. The real catalyst is formed in the initial reaction stage.

The redistribution of fatty acids can be directed or left to operate at random. In the first case, an equilibrium, corresponding to the law of probability, is reached: the fatty acids are redistributed non-selectively throughout the different esters.

Interesterification can also be directed to a certain extent, e.g. by segregation of the newly formed high melting esters from the reaction mixture through controlled crystallisation during interesterification.

Random interesterification is very commonly used, whereas directed interesterification applies to special cases only.

In order to allow the catalytic reaction to proceed, the oil or oil blend needs to satisfy certain quality standards, depending on the catalyst used. When alkaline catalysts are used, care should be taken with water, free fatty acids and peroxides, as they destroy the catalytic activity.

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- combine the properties of mixed oils and fats
Fatty acid neutralisation and drying are therefore necessary prior to the addition of the catalyst.

Drying under vacuum at elevated temperature and prebleaching of the oil also reduce the peroxides, which is beneficial for the catalytic activity. This explains why the feedstock in interesterification is generally neutralised or, in most cases, neutralised bleached oil.

**Catalyst Poisoning**

<table>
<thead>
<tr>
<th>Poison</th>
<th>Type</th>
<th>Level</th>
<th>Catalyst inactivated (kg/ton oil)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Water</td>
<td>0.01%</td>
<td>Na 0.13, CH₃ONa 0.3, NAOH 0.1</td>
</tr>
<tr>
<td></td>
<td>Fatty Acid</td>
<td>0.05%</td>
<td>Na 0.04, CH₃ONa 0.1, NAOH 0.07</td>
</tr>
<tr>
<td></td>
<td>Peroxide</td>
<td>1.0</td>
<td>Na 0.023, CH₃ONa 0.054, NAOH 0.04</td>
</tr>
<tr>
<td></td>
<td>Total</td>
<td></td>
<td>Na 0.193, CH₃ONa 0.454, NAOH 0.11</td>
</tr>
</tbody>
</table>

**Oil Quality**

| Semi-refined (neutralised/bleached) | FFA < 0.05% | Phosphorus < 2 ppm | Moisture < 0.05% | Peroxide value < 0.5 meq/kg | p-Anisidine value < 10 |

**Catalyst**

As already mentioned, the best known catalysts are the alkali metals such as sodium, potassium and their alloys and alkoxides such as sodium methylate or sodium ethoxide. Alkali hydroxides as sodium hydroxide are also used occasionally, but they are less active and therefore require higher temperatures (above 150°C). One of the most popular catalysts used today are sodium methylate or sodium ethylate.

**Reaction conditions**

During the reaction with, for example, sodium methylate, fatty acid methyl esters and fatty acid soaps are formed. Every mole of sodium methylate catalyst added to the oil generally generates one mole of fatty acid methylester as well as one mole of soap.

As a result of this, the mono- and more specifically diglyceride content of the interesterified oil increases, which is not always beneficial to the oil quality.

Nearly all monoglycerides as well as - to a much lesser extent - diglycerides are removed during deodorisation at elevated temperature. Soaps, however, require removal by other physical (adsorption) or chemical means (acid treatment). If this is not done properly, considerable emulsion problems may appear. The addition of the correct amount of catalyst is therefore a critical point during processing.

The reaction can be stopped by deactivation of the catalyst. This can be done by adding water to the solution (wet catalyst deactivation) or by using an acid, usually phosphoric acid or citric acid (dry catalyst deactivation) to convert the soaps back into their free fatty acid state.

Special adsorptive clays like trysil can also be used to stop the reaction and remove the soaps. Typical industrial values
for a random interesterification with sodium methylate catalyst are as follows:

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Interesterification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature °C</td>
<td>70 → 150</td>
</tr>
<tr>
<td>Catalyst conc. %</td>
<td>0.05 → 0.2 (Na-methylate)</td>
</tr>
<tr>
<td>Pressure mbar</td>
<td>&lt; 30 mbar absolute (drying)</td>
</tr>
<tr>
<td>Reaction time min</td>
<td>18 → 90</td>
</tr>
</tbody>
</table>

### Interesterification technology

Industrial interesterification is a batch or a continuous operation. Most modern interesterification units are semicontinuous. The main feature of the semicontinuous process is that each unit operation is carried out in a specifically designed vessel.

In a first stage, caustic soda is dosed slowly to a measured quantity of oil or blend of oils, to neutralise the free fatty acids, and the mixture is heated (120-130°C).

After this, the neutral oil is sprayed slowly in the reaction vessel under reduced pressure (10-30 mbar), and kept there for about 30 min to remove as much water as possible. During drying, the oil is pumped around in order to speed up the drying process.

The oil is subsequently cooled down to about 90°C and the catalyst introduced: some 0.05 to 0.2 % of catalyst are added, as a dry powder or as a slurry mixed with dried oil.

The mixture is intensely mixed for about 30 min. The reaction is visualised by a strong dark coloration of the oil. When the reaction is completed, water or acid is added, to destroy the catalyst and, hence, to stop the reaction.

When acid is used to destroy the catalyst, a small excess is added in order to convert the FA soaps back into free fatty acids.

The mixture as such is subsequently dried, bleached and deodorised.

### Catalyst dosing

Most catalysts are very aggressive and sensitive to moisture, and therefore require very careful handling.

Normally, the catalyst is supplied in preweighed quantities in special small drums and inserted manually into the reactor vessel. Since the vessel operates under reduced pressure, an intermediate slot is installed to prevent air and moisture from entering.

New installations are equipped with an automatic powder dosing system, reducing manual intervention.

### Post - bleaching filters

Bleaching is required after interesterification to remove the dark reaction color of the oil as well as residual soaps and other unwanted components.

The plate and frame filters, which are normally used in the postbleaching process, are more and more replaced by very sophisticated vertical or horizontal tank filters. In the USA, horizontal tank filters are preferred, whereas in Europe, vertical tank filters are very much in use.

Interesterification is a source of considerable oil losses. First of all, those caused by the reaction with the catalyst: free fatty acids (under the form of soaps) (FFA) and fatty acid methyl esters (FAME) are produced.

When they are removed during deodorisation, they cause an additional oil loss of about 20-30% due to oil entrainment in the vapour phase.

During post - bleaching, about 0.3 to 0.4 kg of oil are lost for every kg of bleaching earth added to the oil. About 0.5 to 1% are generally added, which represents a loss of about 1.5 to 3 kg of oil per ton of processed oil.

In the case of interesterification with 0.1% of NaOCH₃ catalyst and a bleaching earth consumption of 0.5%, the overall oil loss is estimated around a minimum of 1.5%, which is high.

In most industrial plants in operation today, however, the effective oil losses during interesterification and post-refining are higher (sometimes up to three times higher) as a result of too much manual handling and improper operation.

An accurate monitoring of the oil quality (moisture, peroxides, residual FFA and phosphorus, mono- and diglyceride content, etc...), as well as a careful operation of the interesterification plant during the successive stages (neutralisation, drying, catalytic reaction, inactivation, post-bleaching and deodorisation) are therefore of the utmost importance to reduce oil losses and, consequently, to improve the profitability of the process.
Physical data of some mixtures of native (SBO) and fully hydrogenated soyabean oil (FHSBO) before and after chemical interesterification.

<table>
<thead>
<tr>
<th>Mixture SBO/FHSBO</th>
<th>90/10</th>
<th>80/20</th>
<th>70/30</th>
<th>60/40</th>
<th>50/50</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Init</td>
<td>Est</td>
<td>Init</td>
<td>Est</td>
<td>Init</td>
</tr>
<tr>
<td>0°C</td>
<td>12.1</td>
<td>9.3</td>
<td>23.1</td>
<td>21.4</td>
<td>34.2</td>
</tr>
<tr>
<td>10°C</td>
<td>12.0</td>
<td>3.0</td>
<td>22.9</td>
<td>12.3</td>
<td>33.6</td>
</tr>
<tr>
<td>20°C</td>
<td>12.1</td>
<td>3.0</td>
<td>22.8</td>
<td>10.0</td>
<td>32.9</td>
</tr>
<tr>
<td>30°C</td>
<td>10.4</td>
<td>1.8</td>
<td>18.8</td>
<td>4.2</td>
<td>29.9</td>
</tr>
<tr>
<td>40°C</td>
<td>8.0</td>
<td>0.9</td>
<td>16.5</td>
<td>2.0</td>
<td>25.1</td>
</tr>
<tr>
<td>50°C</td>
<td>3.4</td>
<td>0.0</td>
<td>10.3</td>
<td>0.0</td>
<td>17.3</td>
</tr>
<tr>
<td>MP°C</td>
<td>52</td>
<td>24</td>
<td>62</td>
<td>51</td>
<td>64</td>
</tr>
<tr>
<td>IV</td>
<td>121.1</td>
<td>107.4</td>
<td>90.8</td>
<td>81.5</td>
<td>69.9</td>
</tr>
</tbody>
</table>

SFC: solid fat content (AOCS serial method) - MP: melting point; IV: iodine value

**Interchem® Management**

**Process Automation**

**Easy management**
Whatever the size of the plant, the various processes used in winterising are easy to manage today, thanks to the rational approach offered by computers and programmable logic controllers.

**Reduction of risks**
Automation serves various purposes, among which we would like to mention the reduction of risks attributable to human mistakes, the obtention of a better and constant quality, superior yields, reduced consumptions and a higher degree of safety.

**Any framework of any network**
The framework of automation networks – to which it is easy to add other digital systems- is so flexible that numerous solutions exist, for both new and old plants. These networks do meet the requirements of all the processes used today.

**Centralised Supervision**

**Permanent follow up**
Centralised supervision is the most efficient tool to permanently follow production.

**Overall view**
The method, using selected softwares, continually offers an overall view of the ongoing activities and of their historical account.

**Reports**
- Analogic reports
- Reports on variables
- Preventive maintenance reports
- Production reports: flowrates, quality controls, etc
Easy to analyze, these reports are the undeniable witness of the complete activity of a process.

**No data unattended**
Central supervision is the ultimate management tool that leaves no data unattended.

For more information on Interchem® technology for your specific process, contact your local Desmet Ballestra office!