

SCI LECTURE PAPERS SERIES  
**CHEMICAL INTERESTERIFICATION – PROCESS  
AND SAFETY MANAGEMENT**

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### **Safety management**

Sodium methylate, commonly referred to as sodium methoxide, is extremely hygroscopic and will react exothermically with atmospheric water vapour to produce sodium hydroxide and methanol. It will also react with moisture on human tissue causing severe damage to vulnerable areas such as eyes or airways, and can lead to skin diseases with even light contact.

Particle size distribution is in the range 10-50 microns, consequently emptying the bags of catalyst should be carried out with the maximum level of containment and the use of a glovebox dispenser and an enclosed dispensing system is strongly advocated (Fig 1). When handling the bags of catalyst, operators should also wear appropriate protective equipment.

The principal suppliers of sodium methylate (methoxide) in the UK, BASF, stress the corrosive nature of the sodium methylate crystals and the dangers of fire/explosion in their storage and handling booklet.

Sodium methoxide is also available in liquid form as a solution in 75% methanol. This option is not recommended. Reactivity is reduced and the extra methanol can enter the reaction, producing very large quantities of methyl fatty acid esters, which will flash off during deodorisation.

Most incidents involving sodium methoxide catalyst stem from the fact that the catalyst looks harmless and is often treated with insufficient caution. One incident involved returning a part used bag to the catalyst store. This action resulted in a small fire that spread to the whole stock, when the fire brigade tried to extinguish the fire with water hoses.

The only safe way to extinguish a minor fire is to smother the effected catalyst with dry sand or similar material. Fullers earth was on at least one occasion found to be effective in extinguishing a sodium methoxide fire in a drum.

Catalyst should be added to the reactor under vacuum without a major ingress of air, and an appropriate method is illustrated (Fig 2).

One of the important elements of process safety is the minimisation of catalyst usage, which reduces the risk as well as the process losses.

## Process losses

During the reaction, and during termination, the catalyst can create a number of undesirable side reactions resulting in the formation of fatty acid methyl esters, soaps and mono- and diglycerides. In stoichiometric terms (based on stearic acid) each 0.1% sodium methoxide can react with the oil to produce:

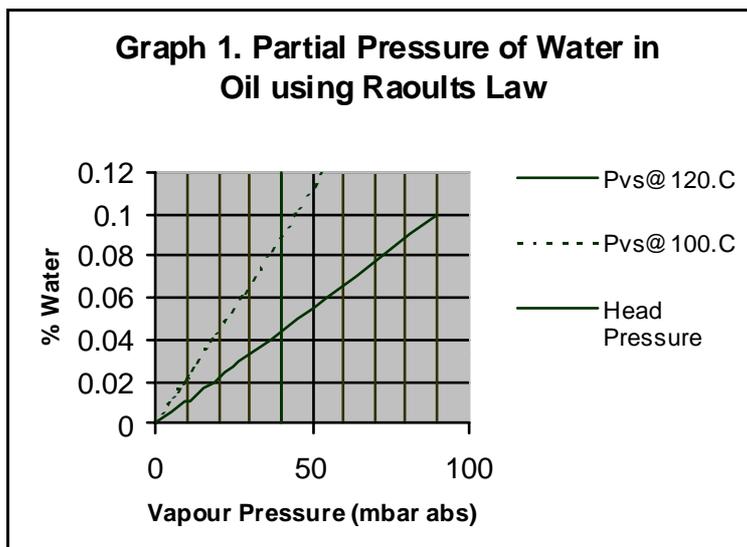
- 0.58% fatty acid methyl ester plus 0.58% soap (or ffa) plus 1.2 - 2.4% monoglyceride and diglyceride

Most of these products will be lost during processing and subsequent deodorisation. This, together with bleaching oil in earth losses, neutral oil losses and other deodorising losses means that with near perfect interesterification using just 0.1% catalyst the losses can be well in excess of 2%.

Sodium methoxide is de-activated by water, ffa or peroxides, leading to much higher usage of catalyst. Water is the biggest problem, as stoichiometrically 0.1% catalyst can be de-activated by 0.033% water. Ideally water should be reduced to below 0.01%, but this has been found by experience to be far more difficult than it appears, due to the low molar concentration of water, leading to correspondingly low partial pressure, as indicated by Raoult's Law Graph 1 shows that at typical bleacher vacuum levels of 40 mbar the equilibrium water content, assuming no oleostatic pressures, would be  $\sim >0.04\%$  at 120°C.

In order to reach below 0.01% the pressure should be reduced to  $<10$  mbar, but the time taken to reach equilibrium in a conventional deep bed reactor will remain a problem.

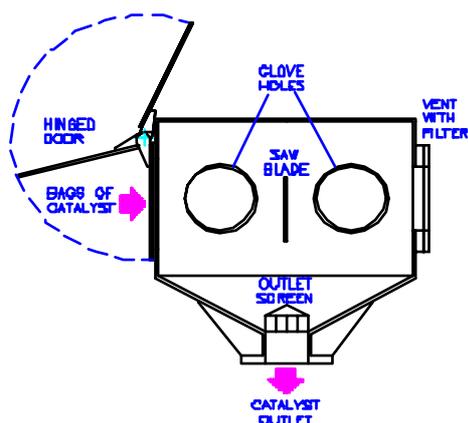
Generally refiners use traditional batch bleachers, operating at  $\sim 40$  mbar to carry out interesterification reactions, and are surprised at the high losses they incur. This is generally associated with excessive use of catalyst.



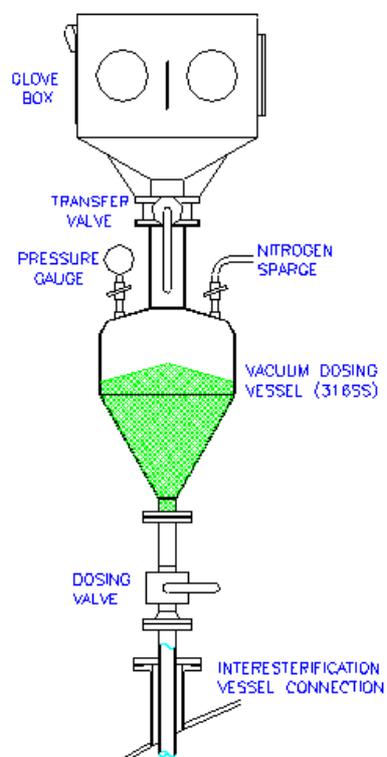
The alternative is to follow the same principle as used in deodorisation, obeying Dalton's law of partial pressures using nitrogen as a carrier gas to remove the water. This method is already used by some refiners, however the amount of nitrogen used is arbitrary and, to the speaker's knowledge, no-one has published a calculation showing the requirement.

In order to remedy this the writer has used the full deodorisation equation as derived by Dijkstra to calculate the quantity of nitrogen to reduce water from 0.04 weight % to 0.01 weight % at 40 mbar and 120°C. The result was a nitrogen requirement of 0.15 % of oil weight. This assumed a vaporisation efficiency factor of 0.5.

These factors have led Ebotec to design a semi-continuous drying system, which will offset the problems of oleostatic pressure and provide optimum vaporisation efficiency, and can be used for batch or continuous interesterification processing.



**Figure 1.** Glove box design



**Figure 2.** Catalyst dosing system design