

Abstract

A process for preparing tri- and tetraoxymethylene glycol dimethyl ether (POMDME_{n=3,4}) by reacting formaldehyde with methanol and subsequently working up the reaction mixture by distillation, comprising the steps of:

- a) feeding aqueous formaldehyde solution and methanol into a reactor and reacting;
- b) feeding the reaction mixture a into a reactive evaporator and separating into a low boiler fraction b1 comprising formaldehyde, water, methanol, methylene glycol, polyoxymethylene glycols, hemiformals, methylal and polyoxymethylene glycol dimethyl ethers (POMDME_{n>1}), and a high boiler fraction b2, and recycling the high boiler fraction b2 into the reactor (step a));
- c) feeding the low boiler fraction b1 into a first distillation column and separating into a low boiler fraction c1 comprising formaldehyde, water, methylene glycol, methanol, hemiformals, methylal, di-, tri- and tetraoxymethylene glycol dimethyl ether (POMDME_{n=2,3,4}), and a high boiler fraction c2, and recycling the high boiler fraction c2 into the reactive evaporator (step b));
- d) feeding the low boiler fraction c1 into a second distillation column and separating into a low boiler fraction d1 and a high boiler fraction d2 consisting of formaldehyde, water, methylene glycol, polyoxymethylene glycols, tri- and tetraoxymethylene glycol dimethyl ether (POMDME_{n=3,4});
- e) feeding the high boiler fraction d2 into a phase separation apparatus and separating into an aqueous phase e1 consisting of formaldehyde, water, methylene glycol and polyoxymethylene glycols, and an organic phase e2 comprising tri- and tetraoxymethylene glycol dimethyl ether (POMDME_{n=3,4});

- f) feeding the organic phase e2 into a third distillation column and separating into a low boiler fraction f1 consisting of formaldehyde, water, methylene glycol and polyoxymethylene glycols, and a high boiler fraction f2 consisting of tri- and tetraoxymethylene glycol dimethyl ether (POMDME_{n=3,4}).