

Experiment 9: Preparation of N,0-carboxymethyl chitosan

Duration: 90 minutes (without drying time).

Equipment: 2 beaker (100 ml), magnetic stirrer, stirring rod, filter, filter rack, filter paper, pH paper, crystallization dish (9 cm), desiccator, water pump. Reagents and materials: chitosan, monochloroacetic acid, sodium hydroxide solution, w(NaOH) = 50 %, acetic acid, w(C₂H₄O₂) = 98 %, acetic acid, w(C₂H₄O₂) = 12 %, ethanol, silica gel blue.

Procedure: 1 g of chitosan is suspended in 50 ml of diluted sodium hydroxide solution. Then 1 g of monochloroacetic acid is added carefully. Now the mixture is stirred for 1 hour. Afterwards the pH value is brought to 5 by addition of acetic acid and the precipitate formed is filtered off. The product is washed with ethanol and finally it is dried in the desiccator over silica gel blue. *Observation:* After stirring for 1 hour a high viscous solution is formed. On addition of acetic acid (pH 5) a flocculent, almost colorless precipitate is formed, which is easily filtered off.

Faults and precautions: Because of the vigorous reaction by addition of chloroacetic acid to sodium hydroxide solution, the experiment has to be performed in the hood and protective gloves have to be worn. If stirring time is shortened, possibly not all of the chitosan has reacted with the monochloroacetic acid. In this case in alkaline medium undissolved chitosan remains. *Waste disposal:* The acetic acidic solution is neutralized and poured down the sink.

