

Syntesis and kinetic study of phenol-formaldehyde resins synthesized in the presence of polyamines

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Abstract

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Protocol

Synthesis of phenolic resin (catalysts)

Step 1.

The amounts of phenol, formaldehyde (47% aqueous solution) and catalyst ie. triethylamine or ammonia solution (25%) or diethylenetriamine or triethylenetetraamine was charged to the reactor resulted from the assumed mole ratio (1.0 : 1.15 : 0.03).

AMOUNT

95.06 g Additional info: Phenol, 99%

AMOUNT

73.48 g Additional info: Formalin, 47%

AMOUNT

3.1 g Additional info: diethylenetriamine, 99%

AMOUNT

4.52 g Additional info: triethylenetetraamine, >97%

AMOUNT

3.04 g Additional info: triethylamine, >99%

REAGENTS

Phenol by [Sigma Aldrich](#)

✓ Formaldehyde solution by Contributed by users

triethylamine T0886 by [Sigma Aldrich](#)

diethylenetriamine D93856 by [Sigma Aldrich](#)

✓ triethylenetetraamine 90460 by Contributed by users

ammonia solution 25% 000000001054280 by [Sigma Aldrich](#)

Synthesis of phenolic resin (catalysts+cocatalysts)

Step 2.

In case of synthesis with used catalyst i.e. triethyleneamine and cocatalysts i.e. diethylenetriamine or triethylenetetraamine the amounts of phenol, formaldehyde (47% aqueous solution) and triethylamine and diethylenetriamine or triethylenetetraamine was charged to the reactor resulted

from the assumed mole ratio (1.0 : 1.15 : 0.03 : 0,005).

AMOUNT

3.04 g Additional info: Triethylamine, >99%

AMOUNT

0.52 g Additional info: Diethylenetriamine > 99%

AMOUNT

0.73 g Additional info: Triethylenetetraamine > 97%

REAGENTS

Phenol by [Sigma Aldrich](#)

✓ Formalin, 47% by Contributed by users

Triethylamine T0886 by [Sigma Aldrich](#)

Diethylenetriamine D93856 by [Sigma Aldrich](#)

Triethylenetetraamine 90460 by [Sigma Aldrich](#)

Step 3.

Process of synthesis

Step 4.

The synthesis was carried out for 5 hours at 75°C. After that the reaction mixture was cooled down to 50°C and its concentration was increased in a laboratory vacuum evaporator to reach the water content of 6 -7%. Then, methanol was added to the concentrated product at a constant amount of 20%. The product resins were analysed for their compositions and physical-chemical properties, like viscosity, non-volatile matter, gelation time.

Kinetic study

Step 5.

The kinetic studies were carried out according to the previous procedure listed above with the following difference: the reaction mixture was sampled at specific time intervals, each sample was immediately placed in the freezing mixture, at approx. -30°C, thereby the chemical reactions were effectively slowed down, after that the concentration of phenol and formaldehyde was determined by HPLC.

The way of calculation of rate constant is enclosed in external links below.

LINK:

<https://drive.google.com/open?id=0BwPhQ7qfvoayenZGMUp2SkhLb0I3SXBHbXU3cWY4cXBxRU5F>