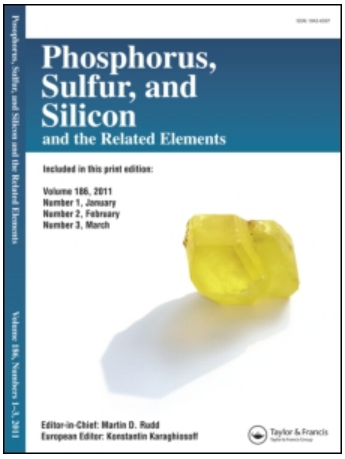

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Synthesis of Phosphorus Containing Polymers by Phase Transfer Catalysis (PTC). II. Experimental Design for Polycondensation in Liquid-Vapor System

Smaranda Iliescu[a]; Ludovic Kurunczi[ab]; Gheorghe Ilia[a]; Gheorghe Dehelean[a]; Lavinia Macarie[a]
[a] Romanian Academy, Institute of Chemistry, Timisoara, Romania [b] University of Medicine and Pharmacy Timisoara, Timisoara, Romania

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Synthesis of Phosphorus Containing Polymers by Phase Transfer Catalysis (PTC). II. Experimental Design for Polycondensation in Liquid-Vapor System

SMARANDA ILIESCU[a], LUDOVIC KURUNCZI[ab], GHEORGHE ILIA[a], GHEORGHE DEHELEAN[a] and LAVINIA MACARIE[a]

[a]*Romanian Academy, Institute of Chemistry, 1900 Timisoara, Romania and*
[b]*University of Medicine and Pharmacy Timisoara, 1900 Timisoara, Romania*

The simultaneous influence of four factors [reaction time (*t*), temperature (*T*), base concentration (*c*), molar ratio of the reagents (*r*)] on the yield (η) and on the inherent viscosity (η_{iv}) of the product has been studied for the liquid-vapor system polycondensation of cyclohexyldichlorophosphonate (CDP) with bisphenol A (BA) by second order central composite circumscribed design. For a reasonable domain of the parameters, [t = 30 ÷ 70 min., T = 20 ÷ 60 °C, c = 1 ÷ 5 mol/l, r = 1 ÷ 3.5 mol(CDP)/mol(BA)], designing the experiments, the η and η_{iv} values where determined in 31 synthesis (7 center points were used). In order to model the response (hyper)surfaces for η and η_{iv}, second order equations with interaction terms were used. Analyzing these model equations, T and c appear to be the most important factors, but acting in opposite way. Higher T resulted in greater yield and inherent viscosity. In the above defined domain the hypersurface of η presents a saddle point. From the sequential inspection of the dependence of η and η_{iv} on the different parameter pairs, using contour plots and 3D-plots [see for example the attached figure for the yield function of factors x2 (T) and x3 (c)] optimum conditions can be deduced for the desired good yield and great inherent viscosity. These are: long reaction time (70 min), high temperature (60 °C), low base concentration (1 mol/l) and high to moderate CDP excess.