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

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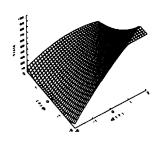
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Synthesis of Phosphorus Containing Polymers by Phase Transfer Catalysis (PTC). II. Experimental Design for Polycondensation in Liquid-Vapor System

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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, $[t=30 \div 70 \text{ min.}, T=20 \div 60 \text{ °C}, c=1 \div 5 \text{ mol/l}, r=1 \div 3.5 \text{ mol}(CDP)/\text{mol}(BA)]$, 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/1) and high to moderate CDP excess.