Polymers, Environment & Sustainable Developments and Opportunities for the Coming Decade

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Workshop 2017

April 30th to May 3rd
Albufeira, Algarve, Portugal

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N. Marques, M. van Duin, A.V. Machado

EVALUATION OF THE ALMOND SHELL OXYPROPYLATION PROCESS TROUGH THE SURFACE RESPONSE METHODOLOGY

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The efficiency of an oxypropylation process depends on several variables and operating conditions, which may not be generalized due to the diverse nature of subtracts. In lignocellulosic biomasses, the content of each fraction (lignin, cellulose and hemicellulose) can differ, as well as, the crystalline organization, which may limit reagent's access to biomass. Also, the hydroxyl content can vary among biomasses; high values demand higher amounts of reactants, namely catalyst content, and more severe reaction conditions. Therefore, owing to biomass variability, selection of the operating conditions for oxypropylation and their optimization is a key issue. In this context, one-factor-at-a-time approaches are commonly used to optimize processes; but it is well-known that optimal operating conditions or interactions between variables cannot be predicted by this simplistic method. Both problems may be overcome by employing the response surface methodology (RSM). In fact, classical methods to optimize the process variables involve changing one variable at a time, keeping the others at fixed levels. This is a laborious and time-consuming method that often does not guarantee the determination of optimal conditions. On the other hand, carrying out experiments with every possible combination of the involved variables is impractical due to the large number of required trials.

In this work, the RSM method was used to optimize the oxypropylation process of a lignocellulosic agro-industrial residue (almond shell). The chosen independent variables were the catalyst content (Cat, X1), expressed as % (w/w, AS-basis) and the almond shell to propylene oxide ratio (AS/PO ratio, X2), expressed in g/ml. The set point temperature was kept constant (160 ºC). The response variables were the hydroxyl index (OH index, Y1), expressed as mg KOH/g, the homopolymer content (HOMO, Y2) expressed as % (w/w, polyol-basis), the unreacted biomass (BIOMASS, Y3), expressed as % (w/w, biomass-basis) and the viscosity (μ, Y4), expressed as Pa.s (20 ºC). Polyol requirements were defined with respect to polyurethane foam preparation (OH index between 300 and 800 and viscosity below 300 Pa.s).

Two different optimization approaches were performed: A1) the minimization of responses Y2 (HOMO) and Y3 (BIOMASS), individually and jointly, and by controlling the restricted responses within the marginal values of Y1 (OH index) and Y4 (viscosity); and A2) the minimization of the responses Y2 (HOMO) and Y3 (BIOMASS) in a multivariable form to produce, jointly, the profiles of all the responses and variables and therefore, provide a global interpretation of the oxypropylation process. Both optimization approaches (A1 and A2) meet equivalent conclusions but the second one revealed to be more informative. In fact, for the evaluation of the oxypropylation process, the patterns of the restricted responses (OH index and viscosity) and variables (Cat and AS/PO) can be found for all the possible minimization solutions of the target responses (HOMO and BIOMASS), providing a much more global interpretation of the process.

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