

Special Research Project Title	Synthesis and Chracterization of Biodegradable Poly (vinyl alcohol)- <i>graft</i> -stearic acid
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Abstract

Poly(vinyl alcohol)-*graft*-stearic acid (PVA-g-SA) copolymers with different degree of substitution (DS) were successfully prepared by one-pot grafting reaction using 1,1'-carbonyldiimidazole (CDI) as a coupling agent, and their structural characteristics were determined by FTIR-ATR and ¹H-NMR spectroscopic techniques. In the synthesis step, the yield and DS of the PVA-g-SA copolymer were found to increase with increasing the reaction time. The suitable reaction time in the coupling reaction step was found to be 24 h at which the yield and DS of the obtained PVA-g-SA product were 57.36 % and 49.57 %, respectively. The study on the influence of solvent pretreatment demonstrated that the yield of the PVA-g-SA was improved from 36.53 % to 57.36 % when predried DMSO was used as a solvent. The decompositions of CDI and SA-imidazolide intermediate caused by the water residue in non-predried DMSO were likely responsible for the lower yield. Furthermore, higher DS values were expected to be obtained when increasing the molar feed ratio of SA to PVA. However, it was found that the DS of the PVA-g-SA product increased from 29.13 % to 49.57 %, and then decreased to 41.67 % when the mole of PVA decreased from 37.5 mmol to 18.75 and 12.5 mmol, respectively. The decrease in the yield could be explained by poor mixing of the reaction mixture due to the increase in the viscosity. The preliminary study on the reaction scalability revealed that increasing the reaction scale by 7 and 15 times resulted in the decrease in the yield from 57.36 % to 52.66 % and 44.82 % respectively and the decrease in the DS value from 49.57 % to 45.37 % and 33.24 % respectively. These decreases could be explained by poor mixing of the reaction mixture and undesired reactions due to the presence of water residue. DSC analysis revealed that the PVA-g-SA copolymer with a DS of 30.05 % had a crystalline melting temperature (T_m) of 50.17 whereas the PVA-g-SA copolymer with a DS of 52.68 % had two T_m suggesting two different types of crystal structure being formed. The PVA-g-SA copolymer with a DS of 30.05 % also had glass transition temperature (T_g) of 26.32 °C while no T_g was observed for the PVA-g-SA copolymer with a DS of 52.68 % and 33.24 %. Regardless of the difference in the DS, these graft copolymers possessed similar solubility, which they were completely soluble in the solvents that have the polarity index between 4 and 2.8 but insoluble in high polar and non polar solvents.

Keywords: coupling reaction / Poly(vinyl alcohol)-*graft*-stearic acid / grafting reaction / 1,1'-carbonyldiimidazole / fatty acids