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ANIONIC POLYMERISATION

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ISOPRENE-STYRENE BLOCK COPOLYMER. THESIS ADVISORS : PRANEE  
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Polymers with high degree of unsaturation are prone to be thermally and oxidatively unstable, especially when experiencing the melt-mixing process. Hydrogenation is one of the practical reactions used to change chemical structure in order to improve thermal properties. In this study, poly(isoprene-*b*-styrene) prepared by anionic polymerisation was hydrogenated by non-catalytic method using *p*-toluenesulphonyl hydrazide (TSH) as a hydrogen releasing agent. The block copolymers having various molecular weights and certain equal block composition (50:50) were synthesised in cyclohexane at 40°C using *n*-BuLi as an initiator. Characterisation of the product by <sup>1</sup>H-NMR showed that the ratio of cis-1,4, trans-1,4 and 3,4-polyisoprene obtained in polyisoprene block was 70:25:5. For the styrene block, it was likely to be atactic polystyrene because the single broad absorption band was observed in range of 1040-1100 cm<sup>-1</sup>. Hydrogenation reaction of the block copolymer was carried out in xylene solution at 135°C using TSH. The mole ratio of TSH to double bond was varied, i.e. 1:1, 2:1, 4:1, and 6:1 as a preliminary study. It was found that the mole ratio of 4:1 is the optimum ratio, giving rise to high percentage of hydrogenation and rather low content of TSH by-product. Percentage of hydrogenation was determined by three techniques, i.e. IR, <sup>1</sup>H-NMR, and Iodine Value. Molecular weight and molecular weight distribution of hydrogenated products were measured using GPC. A slight decrease in molecular weight was found which could be due to degradation caused by the relatively high reaction temperature. From DSC measurement, *T<sub>g</sub>* of the hydrogenated product was found to be increased about 10-20°C above that of the original block copolymers. The thermal stability of hydrogenated products was slightly improved as shown by TGA. It was also found that cis-trans-isomerisation took place during the hydrogenation process. By-product obtained from dissociation of TSH was also investigated. Purification of hydrogenated products was also carried out.