TRANSFORMATION OF SUCROSE
(PREPARATION AND CHARACTERIZATION OF SUCROSE POLYESTERS OF DICARBOXYLIC ACID)

by

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Dedicated to my

Parents

C E R T I F I C A T E

This is to certify that the thesis entitled, "Transformation of Sucrose (Preparation and Characterisation of Sucrose Polyesters of Dicarboxylic Acids)", being submitted by Mr. Sunil Kumar Mattey to the Indian Institute of Technology, Delhi for the award of the Degree of 'Doctor of Philosophy in Chemistry', is a record of bonafide research work carried out by him. Mr. Sunil Kumar Mattey has worked under my guidance and supervision and has fulfilled the requirements for the submission of the thesis which to my knowledge has reached the requisite standard.

The results contained in this thesis have not been submitted, in part or in full, to any other University or Institute for the award of any degree or diploma.

(Dr. J.L. Norula)
Research Supervisor

A C K N O W L E D G E M E N T

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A B S T R A C T

This thesis deals with the Synthesis and Characterization of Poly(sucrose esters). Sucrose (1 mole) was condensed with adipic sebacic and terephthalic acid chlorides/esters (in 2, 4, 6 and 8 moles respectively) by solution, interfacial and transesterification polycondensation methods.

The samples prepared were characterized by end group analysis, density measurements, solubility and chemical resistance studies, viscosity measurements, infrared spectroscopy (both qualitative and quantitative), quantitative nuclear magnetic resonance and elemental analysis. Thermal behaviour was investigated by thermogravimetric analysis in nitrogen and air atmosphere and differential thermal analysis in air.

It was observed that properties of poly(sucrose esters) were much dependant on the method of their synthesis. Transesterification polycondensation was found to be best approach, since it resulted in isolable soluble products. The slow reaction rate of transesterification also helped in monitoring the reaction and isolation of products at various stages. The interfacial

and solution polycondensation were seen to undergo side reactions besides forming insoluble and infusible products.

End group analysis showed decrease in hydroxyl number with increase in composition ratios of the reactants. An increasing composition ratio was also seen to result in an increase of acid number in case of interfacial and solution polycondensation products.

Intrinsic viscosity $[\eta]$ of the samples were found to be decreasing with increasing composition ratios. Among three poly(sucrose esters), poly(sucrose adipates) showed maximum $[\eta]$. Density measurements showed higher values for poly(sucrose terephthalates) as compared to other samples. Density of the poly(sucrose terephthalates) prepared by solution and interfacial polycondensation were found to be increasing with increasing composition ratios, whereas all other samples showed a decreasing pattern. Quantitative IR and NMR studies of poly(sucrose terephthalates) prepared by transesterification polycondensation revealed the mole fraction of acid incorporated into the samples.

Thermal studies (TGA) indicated higher stability for poly(sucrose terephthalates) than poly(sucrose

adipates) and poly(sucrose sebacates). DTA studies showed only exothermic degradation peaks.

C O N T E N T S