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Publisher *Taylor & Francis*

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## International Journal of Polymeric Materials

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713647664>

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**To cite this Article** Mahajan, S. S. , Sabne, M. B. , Gujar, K. B. and Ghatge, N. D.(1985) 'Selectivity of Isocyanate Modified Cellulose Acetate Membranes to Sugars', *International Journal of Polymeric Materials*, 11: 1, 39 – 45

**To link to this Article:** DOI: 10.1080/00914038508078652

**URL:** <http://dx.doi.org/10.1080/00914038508078652>

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# Selectivity of Isocyanate Modified Cellulose Acetate Membranes to Sugars†

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*(Received August 15, 1984)*

Membrane separation processes are becoming increasingly important for dehydration of liquid food in modern food industry. New membranes with better separation characteristics; improved mechanical, chemical and thermal properties are being developed. In the present work membranes cast from isocyanate modified cellulose acetate polymers have been utilized for the separation/concentration of sugars from their aqueous solutions. Some of the characteristics of the modified polymers and membranes have also been reported.

## INTRODUCTION

During recent years, the purification and concentration of industrial liquids by conventional mass separation techniques have been supplemented by membrane separation processes. In many cases, membrane separation processes are faster, more efficient and economical as compared to the conventional separation techniques. The reverse osmosis (RO) membrane separation process has been used successfully with a wide variety of solution systems.<sup>1-4</sup>

The applications of RO membrane separation process for the

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† NCL Communication No. 3588.

concentration of sugarcane juice and fruit juices is of both scientifically and technically important in recent years. The present paper describes some of the characteristics of the membranes prepared from recently modified cellulose acetate polymers<sup>5-6</sup> for the separation/concentration of sugars such as glucose, sucrose and fructose (normally present in fruit juices) in aqueous solutions. Glucose, sucrose and fructose used in the present study were of commercial grade.

## EXPERIMENTAL

Cellulose acetate (CA), a product of M/s Mysore Acetate, India, having 2.466 degree of substitution was partially reacted with phenyl, propyl and butyl isocyanates separately as per the procedure reported earlier.<sup>5,6</sup> The modified CA polymers thus obtained were labelled as CANCO (0.05), CAPRO (0.05) and CABNCO (0.05) respectively. The numerical number in the bracket indicates the molar quantity of each isocyanate used during modification. The characteristics of these polymers are shown in Table I. The molecular weights of the polymer samples were determined at  $30 \pm 1^\circ\text{C}$  in acetone by viscosity method.<sup>7</sup> The tensile strength measurements were made on Scott Tensile machine according to ASTM Designation D 882-52T. The percent nitrogen in modified polymers has been estimated by Dumas method. The total degree of substitution has been obtained from the original degree of substitution of CA and degree of substitution of urethane groups in the modified polymers. The degree of substitution of urethane groups has been determined by using the following equation.

$$\frac{14x}{265.4 + xM} = \frac{y}{100}$$

where

- $M$  = molecular weight of isocyanate used.
- $x$  = degree of substitution of urethane.
- $y$  = % nitrogen estimated.

The apparent total percent acetyl determined has been compared with the value computed from the known degree of substitution of

TABLE I  
 Characteristics of isocyanate modified cellulose acetate polymers and cellulose acetate

Sample no.	Sample	Softening point °C	[ $\eta$ ] Intrinsic viscosity dL/g	Molecular weight	Tensile strength Kg/cm <sup>2</sup>	% Nitrogen	degree of substitution	Apparent <sup>b</sup> total % acetyl	% Acetyl <sup>a</sup>
1	CANCO (0.05)	238-240	1.55	64 800	187	0.99	2.67	40.6	41.9
2	CAPRO (0.05)	247-250	1.46	61 000	145	1.08	2.68	40.8	39.0
3	CABNCO (0.05)	243-245	1.55	64 800	140	0.91	2.65	40.1	41.2
4	Cellulose acetate (CA)	242-243	1.4	57 800	105	—	2.466	—	39.9

<sup>a</sup> As per ASTM method.

<sup>b</sup> Calculated from total degree of substitutions.

acetyl and urethane in the modified CA. This computation was made as given below.

$$\text{Apparent total \% acetyl} = \frac{100}{265.4 + xM} \times [2.466 + x]43$$

where

$M$  = molecular weight of isocyanate

$x$  = degree of substitution of urethane

Percent acetyl of the polymers has been determined by ASTM Designation D 871-61T.

Membranes were cast in the form of flat sheet as per the method described by Loeb and Sourirajan.<sup>8</sup> The membranes thus obtained were annealed at 88°C for 15 minutes and were preserved in 0.2% formalin solution. The membrane characteristics are given in Table II. Specific water content of the membranes have been determined by the method reported by Ferry.<sup>9</sup> Membrane constant and average pore diameter were determined from pure water permeability of the membranes.

The selectivity of these membranes for glucose, sucrose and fructose solutions has been studied on a laboratory reverse osmosis unit at ambient temperature and operating pressure of 600 psi. The percent sugar rejection has been evaluated from the measurement of concentration of feed and permeate solutions by JASCO DIP-181 Digital Polarimeter (Japan).

TABLE II

Characteristics of the modified cellulose acetate membranes and cellulose acetate membranes

Sample no.	Sample	Pure water permeability gfd	Specific water content g/cm <sup>3</sup>	Membrane constant × 10 <sup>5</sup> g/cm <sup>2</sup> s Atm	Av. pore diameter A°
1	CANCO (0.05)	11	0.63	1.20	22.80
2	CAPRO (0.05)	11	0.60	1.20	22.80
3	CABNCO (0.05)	11	0.58	1.20	23.0
4	Cellulose acetate	14	0.70	1.50	23.80

TABLE III  
Selectivity of modified cellulose acetate and cellulose acetate membranes to sugars

Sugar	CANCO (0.05)			CAPRO (0.05)			CABNCO (0.05)			Cellulose acetate		
	Feed % conc.	Flux gfd	% Rej.	Feed % conc.	Flux gfd	% Rej.	Feed % conc.	Flux gfd	% Rej.	Feed % conc.	Flux gfd	% Rej.
Glucose	1.5	5.6	97.5	1.5	5.6	96	1.5	5.5	96	1.5	6.0	95
	3.0	4.0	97.2	3.0	4.5	96.5	3.0	4.5	95	3.0	5.0	94
	5.0	3.4	97.0	5.0	4.0	95.2	5.0	4.0	95.8	5.0	4.0	92.5
Sucrose	1.5	5.7	97.5	1.5	5.6	96	1.5	5.6	96.5	1.5	6.0	95
	3.0	4.2	97.0	3.0	4.6	96	3.0	4.6	96.0	3.0	4.8	95
	5.0	3.6	97.0	5.0	4.0	95	5.0	3.8	95.5	5.0	4.0	95
Fructose	0.5	5	98	0.5	5.8	97.2	0.5	6	96.5	0.5	6	94.5

Operating pressure—600 psi.  
Operating temperature—23–25°C.  
Membrane area—18.6 cm<sup>2</sup>.

## RESULTS AND DISCUSSION

The modified polymers exhibit higher tensile strength as compared to the parent cellulose acetate. In all the modified polymers the apparent total percent acetyl values calculated from percent nitrogen are in good agreement with the experimentally determined percent acetyl values. Membrane constant and average pore diameter of these membranes are lower as compared to the values obtained from cellulose acetate membrane. The specific water content values are also lower than the specific water content in cellulose acetate membrane. The lower value of specific water content is an indication of reduced water flux which has also been observed experimentally.

The selectivity data for glucose, sucrose and fructose solutions has been summarized in Table III. The selectivity of these membranes to glucose and sucrose has been studied for three different concentrations. Decline in the flux has been observed as the feed concentration increases from 1.5 to 5% in both the cases with negligible deviation in percent sugar rejection. In the case of fructose solution the selectivity has been studied only for one concentration since at higher concentration very much reduced flux was observed. The probable reason for this reduced flux may be the deposition of coloured material from fructose itself.

Few attempts have also been made to concentrate clarified sugarcane juice obtained from M/s Yeshwant Sahakari Sakhar Karkhana, Pune, India, however, the initial experiments are not satisfactory as the flux decreases rapidly due to the deposition of colour and waxy material on the membrane surface. Efforts are being made to overcome these difficulties.

## CONCLUSIONS

The percent sugar rejection and flux rates of the membranes prepared from the modified cellulose acetate polymers are at par with the cellulose acetate membrane prepared in the present study. These membranes might find applications in concentration of fruit juices.

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