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ORIGINAL ARTICLE

Effect of cation exchange resin treatment and addition on sugar as anti-caking agent on retention of nutritional and sensory quality of lemon juice powder during storage

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Abstract Lemon juices clarified with enzymatic treatment with and without cation exchange resin treatment were concentrated to 60^{o} Brix in a vacuum evaporator and converted into powders by foam mat drying technique. Powders obtained from cation exchange resin treated juice were better in quality with respect to acidity, glucose, fructose, sugars, and ascorbic acid contents as compared to those prepared form non treated juice. Further, during 9 months storage, the powders suffered slight loss of acidity, and increase in reducing sugars i.e. glucose and fructose and considerable loss (31–55%) in vitamin C contents. Storage conditions did not bring about any significant change in the ash and hesperidin content of the product. But some losses were registered in the total phenols (23.69%) and sensory quality (from 7.72 to 7.26) of the powders. Further, the powders prepared from cation exchange resin treated juice and those pulverized with cane sugar suffered overall lesser changes in most of the quality parameters during 9 months of storage, thus indicating that, the treatment of lemon juice with cation exchange resin is beneficial for better initial product quality and pulverization of prepared powder with cane sugar is beneficial in reducing the hygroscopicity and retention of quality in a better way.

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Introduction

Citrus products undergo browning and suffer quality loss due to enzymatic or non-enzymatic reactions during processing and storage. Browning of citrus products during heat processing as well as storage has been recognized as a major problem faced by processors. Maintaining the product at low temperature has been and still is the only means to reduce colour and flavour deterioration of processed citrus juice, concentrates and powders for long term storage. But, as soon as the temperature increases during storage, the rate of these deteriorative reactions increases remarkably. The application of heat is almost indispensable during processing, which causes browning and colour deterioration in fruit juice.

The removal of browning substrates thus seems to be a better option to reduce browning and quality deterioration during processing and storage of fruit products, although a compromise is to be made for the removal of one or a few of the nutritional components. The removal of one of the browning substrate i.e. amino acids from lemon juice by use of cation exchange resin treatment has been reported to reduce non-enzymatic browning in finished products during preparation and storage(Sharma et al. 2004a, 2006), but the implications of cation exchange resin treatment on retention of nutritional and sensory quality of lemon juice powder during storage is still scanty in literature. Further, all the fruit juice powders have a property of formation of cake as soon as they are exposed to air. This can be reduced by making use of various anticaking agents. The efficacy of various anticaking agents were evaluated by the authors in their earlier studies and it was found that pulverization of juice powder with powdered cane sugar is very effective for reducing caking and hygroscopicity of the product (Sharma et al. 2002).

Lemon juice is consumed primarily for its high acid and vitamin C contents and not for its amino acid contents. The sugars and amino acids, although present in small amounts in lemon juice, cause considerable browning, colour deterioration, nutritional quality loss and reduce the aesthetic appeal of the product. Therefore, one of the browning reaction substrates could be removed from the juice so that it is not available for reacting with other substrates. Keeping the above facts in view, the present investigations were carried out to evaluate the effect of removal of amino acids (a browning reaction substrate) by cation exchange resin treatment from lemon juice and pulverization of juice powder with cane sugar (for reduction of caking), on nutritional and sensory quality of lemon juice powder during 9 months storage.

Materials and methods

Hill Lemon (*Citrus pseudolimon* Tan.) fruits were harvested at optimum maturity from local orchards in district Sirmour, Himachal Pradesh, India. Fruit juice was extracted by using a semi-automatic motor operated rosing machine (Bajaj Maschinen, Pvt. Ltd., New Delhi, India), after through sorting and washing and was strained through muslin cloth and collected in a stainless steel container to make a homogenous lot. The juice was then heat pasteurized at 90° C for 10 s and preserved in 500 ppm SO_2. The preserved juice was clarified by using "Pectinase CCM" enzyme (Biocon India Ltd., Bangalore, India) at 0.2% for 2 h at 50±2°C (Sharma et al. 2001a). 228 ml of enzymatically clarified juice was passed under gravity through cation exchange resin, Dowex-50 W (Fluka Chemie Gmbh, Switzerland), packed in a glass column (3 cm internal dia.) upto a height of 5.5 cm. Treated juice was collected in a beaker. Column was washed after use with 0.2 N HCl solution (three to four times the volume of resin) and regenerated with 0.2 N NaOH solution (two to three times the volume of resin) with EDTA (AOAC 1995). Excess of alkali was removed by washing cation exchange resin with 3–4 volumes of distilled water and dried under suction at room temperature[1]. Properly washed and regenerated column was repeatedly used for separation of amino acids without any practical loss in its activity. Cation exchange resin treated juice of different lots was pooled together and used for preparation of concentrates. Both types of juices (i.e. cation exchange resin treated and untreated) were converted into concentrates of 60° Brix using a rotary vacuum evaporator (Jain Scientific Glass Works, Ambala, India) at 50±2°C under 28±2" Hg vacuum[1](Sharma et al. 2001b).

The concentrates of 60°Brix prepared from both treated and untreated juices were mixed with carboxymethyl cellulose @ 2%, cooled to a temperature below 5°C followed by whipping in a household mixer for 2–3 min to yield a thick foam which was spread on stainless steel trays (30×20 cm, with a tray load of 100 g per tray) in a thin layer (3–5 mm) and dried in a mechanical dehydrator (55±2°C) for 7 h followed by finishing in a vacuum oven (50±2°C and 25±2" Hg vacuum) for 1.5 h to a constant moisture content of 5–6% (Sharma et al. 2004b, 2005). The dried foam was then scrapped off from the trays and ground in spice grinder to a fine powder. In order to reduce the hygroscopicity of the lemon juice powders, powdered sugar @ 50% was added as anti-caking agent, juice powders prepared from both treated and untreated juices.

Hill lemon juice powders prepared from both treated and untreated juices, with and without addition of 50% powdered sugar, as anti-caking agent (Sharma et al. 2002) were packed in aluminium laminated pouches and stored at ambient temperatures (12.20–31.77°C) for a period of 9 months and analysed periodically for any changes in quality. The flow- diagram of the experimental unit operations has been given in Fig. 1.

Standard analytical procedures were followed to estimate titratable acidity (Ranganna 1997), glucose, fructose, and total sugars (Ting and Deszyck 1960), ascorbic acid (Ranganna 1997), total phenols (Thimmaiah 1999) and flavoniods (Davis 1947). Minerals were determined gravimetrically and expressed as "ash" per cent w/w (AOAC 1984).

For sensory evaluation of lemon juice powders at different storage intervals, instant lemon beverage (powdered) containing 5 g juice powder and 130 g sugar (for preparation of 1 l of RTS beverage) were prepared (Sharma et al. 2001a). The prepared beverages were evaluated for sensory quality based on colour, taste, flavour, body on a 9 point hedonic scale (Amerine et al. 1965).

The data on chemical characteristics were analyzed statistically by completely randomized design (Cochran and Cox 1967), with the factors 2×2×4×3, where 2 indicates the cation exchange resin treatments with two levels (untreated or control juice and treated juice), 2 the use of anticaking agent i.e. sugar with two levels (with addition and without addition of cane sugar), 4 the storage periods with four levels (initial i.e. at the time of product preparation, 3 months, 6 months and 9 months) and 3 the number of analyses carried out (replications). The data pertaining to sensory evaluation were analyzed according to randomized block design.

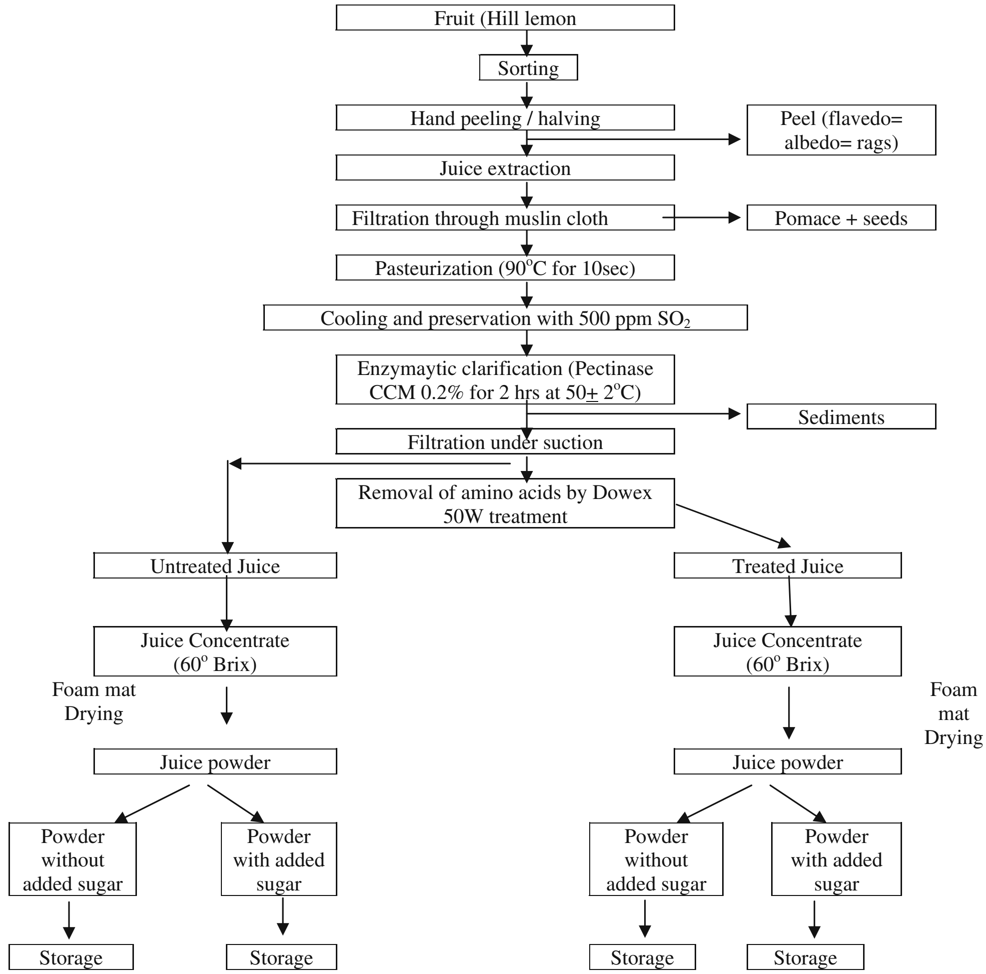

Fig. 1 Diagrammatic representation of the unit operations used for the experiment

Results and discussion

The mean titratable acidity of powders packed with and without added sugar (as anti caking agent) were 27.99 and 56.31% respectively. Considerable lower contents of acidity of powder containing 50% sugar were due to lesser quantities of actual juice powder in it. Further, during 9 months storage, the acidity decreased slightly from 42.22% to 42.07% (mean values), thus, exhibiting a net loss of mere 0.35%. The percent decline in titratable acidity of the powders during 9 months storage were also slightly lesser in those prepared from treated juice (0.32%) as compared to the untreated counterparts (0.37%) thus indicating better retention of quality in the product prepared from cation exchange resin treated juice. Slightly higher amounts of acidity (mean 43.91%) were recorded in powder prepared from treated juice as compared to those prepared from untreated juice (40.38%) which was due to the higher contents of acidity (dwb) in the treated juice than that of the untreated juice (Table 1).

Glucose and fructose contents of lemon juice powders prepared from untreated and treated juices were 9.26 and 10.03% and 9.87 and 10.67% respectively. However, the values in the powders packed without and with added sugar were 12.71 and 13.75 and 6.42 and 6.95% (dwb) respectively. During 9 months storage the powders prepared from treated juice suffered only 0.30 and 0.38% change in their glucose and fructose contents while these were as high as 0.33 and 0.70% in the powders prepared form untreated juice. Therefore the retention of quality was better in the production prepared from treated juice (Table 1).

Further, the mean reducing sugars in lemon juice powders prepared from untreated and treated juices were 19.29 and 20.55% respectively while that of the powders without and with added sugar were 26.46 and 13.38% respectively. During 9 months storage, the values for reducing sugars increased from 19.87 to 19.96% respectively, thus experiencing an increase of about 0.45% on dry weight basis. The changes in reducing sugars were less in powders prepared from treated lemon juice (0.34% increase) as compared to those prepared from untreated juice (0.62% increase) (Table 2).

Contrary to the changes in glucose, fructose and reducing sugars, the non-reducing and total sugars experienced some decline in their contents during storage. The mean non-reducing and total sugars in powders prepared

Table 1 Effect of cation exchange resin treatment and addition of sugar (as anti-caking agent) on the titratable acidity, glucose and fructose contents of lemon juice powders during storage

Storage intervals (months)	Anti-caking agent used[a]	Titratable acidity (%)				Glucose (%)				Fructose (%)			
		Untreated	Treated[b]	Mean	Mean (A)	Untreated	Treated[b]	Mean	Mean (A)	Untreated	Treated[b]	Mean	Mean (A)
Initial	No	54.0	58.8	56.4	56.3	12.3	13.1	12.7	12.7	13.3	14.2	13.7	13.8
	Yes	26.9	29.2	28.0	28.0	6.2	6.6	6.4	6.4	6.7	7.1	6.9	7.0
	Mean	40.5	44.0	42.2		9.2	9.9	9.5		10.0	10.6	10.3	
3	No	54.0	58.7	56.3		12.3	13.1	12.7		13.3	14.2	13.7	
	Yes	26.9	29.2	28.0		6.2	6.6	6.4		6.7	7.2	6.9	
	Mean	40.4	43.9	42.2		9.3	9.9	9.6		10.0	10.7	10.3	
6	No	53.9	58.7	56.3		12.3	13.1	12.7		13.3	14.2	13.8	
	Yes	26.8	29.1	28.0		6.2	6.6	6.4		6.8	7.2	7.0	
	Mean	40.4	43.9	42.1		9.3	9.9	9.6		10.0	10.7	10.4	
9	No	53.9	58.6	56.2		12.3	13.1	12.7		13.4	14.2	13.8	
	Yes	26.8	29.1	27.9		6.3	6.6	6.4		6.8	7.2	7.0	
	Mean	40.3	43.8	42.1		9.3	9.9	9.6		10.1	10.7	10.4	
	Mean (T)	40.4	43.9			9.3	9.9			10.0	10.7		
		CD 0.05				CD 0.05				CD 0.05			
Treatment (T)		0.01				0.02				0.02			
Anti-caking agents (A)		0.01				0.02				0.02			
Storage intervals (S)		0.01				0.03				0.02			
T x S		NS				NS				NS			
A x S		0.02				NS				NS			
T x A x S		NS				NS				NS			

Values expressed on moisture free basis

[a] Anti-caking agent used is 50% powdered cane sugar (w/w basis)

[b] Cation exchange resin (Dowex 50W) treated

Table 2 Effect of cation exchange resin treatment and addition of sugar (as anti-caking agent) on the reducing, non-reducing and total sugars of lemon juice powders during storage

Storage intervals (months)	Anti-caking agent used[a]	Reducing sugars (%)				Non-reducing sugars (%)				Total sugars (%)			
		Untreated	Treated[b]	Mean	Mean (A)	Untreated	Treated[b]	Mean	Mean (A)	Untreated	Treated[b]	Mean	Mean (A)
Initial	No	25.6	27.3	26.4	26.5	1.9	2.3	2.1	2.0	27.6	29.7	28.6	28.6
	Yes	12.9	13.7	13.3	13.4	49.0	49.2	49.1	49.0	64.5	65.6	65.0	65.0
	Mean	19.2	20.5	19.9		25.5	25.8	25.6		46.0	47.6	46.8	
3	No	25.6	27.3	26.5		1.8	2.2	2.0		27.5	29.6	28.6	
	Yes	13.0	13.8	13.4		48.9	49.2	49.0		64.4	65.5	65.0	
	Mean	19.3	20.5	19.9		25.4	25.7	25.5		46.0	47.6	46.8	
6	No	25.6	27.3	26.5		1.8	2.2	2.0		27.5	29.6	28.5	
	Yes	13.0	13.8	13.4		48.8	49.1	49.0		64.4	65.5	64.9	
	Mean	19.3	20.6	19.9		25.3	25.6	25.5		45.9	47.5	46.7	
9	No	25.7	27.3	26.5		1.7	2.2	1.9		27.4	29.6	28.5	
	Yes	13.0	13.8	13.4		48.7	49.1	48.9		64.3	65.5	64.9	
	Mean	19.3	20.6	20.0		25.2	25.6	25.4		45.9	47.5	46.7	
	Mean (T)	19.3	20.5			25.3	25.7			46.0	47.6		
		CD 0.05				CD 0.05				CD 0.05			
Treatment (T)		0.01				0.016				0.010			
Anti-caking agents (A)		0.01				0.016				0.010			
Storage intervals (S)		0.02				0.023				0.014			
T x S		0.03				0.033				0.020			
A x S		NS				0.033				0.020			
T x A x S		0.03				0.047				0.028			

Values expressed on moisture free basis

[a] Anti-caking agent used is 50% powdered cane sugar

[b] Cation exchange resin (Dowex 50W) treated

from untreated and treated lemon juices were 25.33 and 45.95% and 25.67 and 47.56% (dwb) respectively. Further, the corresponding contents of powders stored without and with added sugars were 2.00 and 28.56% and 49.00 and 64.95% respectively on dry weight basis. The storage of powders for 9 months brought about 0.82% and 0.30% decrease in non-reducing and total sugar contents. However, the loss of total sugars was considerably less (only 0.19%) in the powders prepared from treated juice as compared to those from untreated juice (0.39%). The powders stored without added sugar also suffered less loss (0.23%) as compared to those stored with added sugar (0.45%).

Minor increase in reducing sugars of powders during storage might be attributed to the inversion of sucrose and other polysaccharides into reducing sugars. Slightly higher changes in reducing sugars in powders with added sugar was probably due to the presence of higher amounts of sucrose in them, thus causing more inversion. The decline in non-reducing sugars of juice powders during storage might be attributed to their inversion into reducing sugars by hydrolysis in the presence of acids, while that of total sugars was probably due to their involvement in Maillard reactions to form HMF and brown pigments. Further, the lesser changes occurring during storage of powders prepared from treated juice were probably due to the lack of sufficient amino acids to react with sugars during the course of browning reactions.

The mean contents of ascorbic acid in the powders prepared from untreated and treated lemon juice were 99.93 and 158.90 mg % respectively while, that of those stored without and with added anti-caking agent (50% sugar) were 174.60 and 84.23 mg % respectively (Table 3). During 9 months storage of juice powders, about 41.70% reduction was observed in the ascorbic acid contents, with the mean values decreasing from initial 164.74 mg % to 96.05 mg %. However, the loss was only 31.86% in powders prepared from treated juice as compared to 55.31% loss occurring in the powders prepared from untreated juice. The loss of ascorbic acid during storage might be due to their degradation leading to the formation of furfural and brown polymers. The lesser loss in the ascorbic acid contents of the juice powders prepared from treated juice than that of those prepared from untreated juice might be attributed to the presence of low levels of amino acids available to react with ascorbic acid of powder (Sharma et al. 2009).

The mean total phenols in powders prepared from untreated and treated juices were 21.26 and 24.20 mg % respectively, while those stored without and with added sugar were 30.27 and 15.19 mg % respectively. The storage of powders brought about a reduction of about 23.69% in the total phenols with the contents decreasing from initial level of 26.25 mg % to 20.03 mg % after 9 months of storage, thus registering a net loss of 23.70%. However, these losses were only 12.08% in the powders prepared from treated juice as against the high levels of 34.80% in those prepared from untreated juice. Thus, the cation exchange resin treatment of the lemon juice was also observed to be successful in reducing the losses in total phenols of the products.

The mean ash contents in the powders prepared from untreated and treated lemon juices were 0.833 and 0.777% on dry weight basis, while that of those stored without and with added sugar were 1.069 and 0.540% respectively. The 9 months storage of powders did not bring about any significant changes in the ash contents with their mean values varying between 0.802 and 0.808%. The reduction of phenols during storage might be due to their involvement in browning reactions (Cilliers and Singleton 1989). Slightly lower values of ash in the powders prepared from treated juice might be attributed to the removal of metal ions from the juice during cation exchange resin treatment.

The naringin and hesperidin contents of powders prepared from untreated and treated juices were 75.13 and 66.99 mg % and 43.70 and 38.99 mg % respectively while that of those stored without and with added sugar were 79.54 and 70.80 mg % and 39.29 and 35.18 mg % respectively (Table 4). However the storage of powders did not bring about any significant changes in the flavonoid contents with the respective mean values of naringin and hesperidin varying between 59.45 and 59.60 mg % and 52.90 and 53.08 mg % on dry weight basis. However, there was only 0.136% loss in the naringin contents of the powders prepared from treated juice, during 9 months storage, but the losses were as high as 0.036% in those prepared from untreated juice. Considerably lower contents of flavonoids recorded in powders prepared from untreated and treated juice throughout the storage period might be attributed to their initial differences in the respective juices. Slight decline in the sensory scores of the powders might be due to darkening of their colour during storage.

Sensory evaluation of the powders on 9 point hedonic scale indicate that the powders were acceptable throughout the storage period, although a slight but steady decline in the sensory score was observed during the 9 months storage of the powders. Further, the powders prepared from treated juice were rated superior with the mean scores of 7.65 as compared to their counterparts with the mean sensory score of 7.35 only. Similarly, the powders packed with 50% sugar as anti caking agent (7.55) were also rated superior to its counter part (7.45) on the basis of sensory evaluation.

Therefore, it appears that the treatment of lemon juice with cation exchange resin was effective in reducing quality loss to a considerable extent in lemon juice powders. Packing of the highly hygroscopic powders after addition of 50% sugar as anti caking agent was also effective in

Table 3 Effect of cation exchange resin treatment and addition of sugar (as anti-caking agent) on the ascorbic acid, total phenols and minerals (ash) of lemon juice powders during storage

Storage intervals (months)	Anti-caking agent used[a]	Ascorbic acid (mg %)				Total phenols (mg %)				Minerals as Ash (%)			
		Untreated	Treated[b]	Mean	Mean (A)	Untreated	Treated[b]	Mean	Mean (A)	Untreated	Treated*	Mean	Mean (A)
Initial	No	184.6	255.5	220.1	174.6	35.9	34.3	35.1	30.3	1.11	1.04	1.07	1.07
	Yes	91.8	127.0	109.4	84.2	17.8	17.1	17.4	15.2	0.57	0.52	0.54	0.54
	Mean	138.2	191.3	164.7		26.8	25.7	26.3		0.84	0.78	0.81	
3	No	148.6	225.4	187.0		28.5	33.0	30.8		1.11	1.04	1.07	
	Yes	68.8	108.8	88.8		14.7	16.5	15.6		0.57	0.52	0.54	
	Mean	108.7	167.1	137.9		21.6	24.8	23.2		0.84	0.78	0.81	
6	No	126.3	198.2	162.2		25.2	31.8	28.5		1.10	1.04	1.07	
	Yes	55.8	95.7	75.7		13.0	15.9	14.4		0.56	0.52	0.54	
	Mean	91.1	146.9	119.0		19.1	23.8	21.4		0.83	0.78	0.80	
9	No	83.1	175.2	129.1		23.5	30.1	26.8		1.10	1.04	1.07	
	Yes	40.5	85.5	63.0		11.6	15.0	13.3		0.56	0.52	0.54	
	Mean	61.8	130.3	96.0		17.5	22.6	20.0		0.83	0.78	0.80	
	Mean (T)	99.9	158.9			21.3	24.2			0.83	0.78		
		CD 0.05				CD 0.05				CD 0.05			
Treatment (T)		1.61				2.05				0.010			
Anti-caking agents (A)		1.61				2.05				0.010			
Storage intervals (S)		2.27				2.90				NS			
T x S		3.21				4.11				NS			
A x S		3.21				NS				NS			
T x A x S		NS				4.18				NS			

Values expressed on moisture free basis

[a] Anti-caking agent used is 50% powdered cane sugar (w/w basis)

[b] Cation exchange resin (Dowex 50W) treated

Table 4 Effect of cation exchange resin treatment and addition of sugar (as anti-caking agent) on the flavonoids and sensory quality of lemon juice powders during storage

Storage intervals (months)	Anti-caking agent used[a]	Naringin (mg %)				Hesperidin (mg %)				Sensory quality			
		Untreated	Treated[b]	Mean	Mean (A)	Untreated	Treated[b]	Mean	Mean (A)	Untreated	Treated[b]	Mean	Mean (A)
Initial	No	100.5	58.7	79.6	79.5	89.6	52.2	70.9	70.8	7.61	7.82	7.72	7.45
	Yes	50.0	29.2	39.6	39.3	44.6	26.0	35.3	35.2	7.63	7.82	7.73	7.55
	Mean	75.3	43.9	59.6		67.1	39.1	53.1		7.62	7.82	7.72	
3	No	100.5	58.7	79.6		89.5	52.1	70.8		7.39	7.69	7.54	
	Yes	49.8	29.2	39.5		44.5	25.9	35.2		7.52	7.73	7.63	
	Mean	75.1	43.9	59.5		67.0	39.0	53.0		7.46	7.72	7.58	
6	No	100.4	58.7	79.5		89.5	52.1	70.8		7.21	7.55	7.38	
	Yes	49.8	27.5	38.6		44.5	25.9	35.2		7.33	7.63	7.49	
	Mean	75.1	43.1	59.1		67.0	39.0	53.0		7.27	7.59	7.43	
9	No	100.3	58.6	79.5		89.4	52.0	70.7		6.91	7.42	7.17	
	Yes	49.7	29.1	39.4		44.4	25.8	35.1		7.19	7.51	7.35	
	Mean	75.0	43.9	59.5		66.9	38.9	52.9		7.06	7.47	7.26	
	Mean (T)	75.1	43.7			67.0	39.0			7.35	7.65		
		CD 0.05				CD 0.05				CD 0.05			
Treatment (T)		0.07				0.30				0.02			
Anti-caking agents (A)		0.07				0.30				0.02			
Storage intervals (S)		0.09				NS				0.02			
T x S		NS				NS				0.02			
A x S		NS				NS				0.03			
T x A x S		NS				NS				0.03			

Values expressed on moisture free basis

[a] Anti-caking agent used is 50% powdered cane sugar (w/w basis)

[b] Cation exchange resin (Dowex 50W) treated

reducing hygroscopicity and retention of sensory quality of the product during 9 months storage.

Conclusion

Treatment of lemon juice with cation exchange resin “Dowex 50W” before concentration is very much effective in producing a product of better quality and retention of its quality during 9 months storage even at room temperatures. Since, most of the lemon juice based processed products are prepared only after addition of sugar, the powder which is very hygroscopic in nature, can be easily stored by pulverization with powdered cane sugar for reducing caking and retention of better sensory quality during storage. The technology can also be used successfully extended for storage of similar hygroscopic powders.

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