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# PRESERVATION OF CITRUS FRUITS WITH FUNGICIDAL WAX EMULSION

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A fungicidal wax emulsion (Furitex) developed in these Laboratories was examined in detail with regard to its suitability for applying to oranges for extending their storage life. Changes in moisture, total sugar and reducing sugar contents, brix, acidity, vitamin C and respiration rate were determined after treating the fruits with Fruitex, alongwith the control (untreated) fruits. These were found significantly different from those of the treated lots and demonstrated the delaying effect of the ripening process in Fruitex-treated lots. Spoilage of fruits due to mould growth was found to be less in treated lots as compared with untreated lots. Organoleptic evaluation of fruits showed no significant difference between appearance, taste and differently treate fruits.

Citrus fruits are a major fruit crop of West Pakistan, the annual production in West Pakistan being 4,60,000 tons.<sup>1</sup> A large crop of fruit is spoiled due to desiccation and mould growth. The situation is further deteriorated due to picking and transportation. Wax emulsions have been used successfully in other countries<sup>2-4</sup> for extending the storage life of citrus fruits. The well-known composition rely on carnauba wax and certain germicide/bactericides, both of which are not available in the country. Research, therefore, was undertaken at these laboratories to develop wax emulsions based entirely on indigenous materials and the laboratories have been successful in developing a product<sup>5</sup> and which has now being marketted under the trade name of Fruitex.

## **Material and Methods**

Six hundred 'maltas' (sweet oranges) purchased from the local market were water-washed and airdried. They were divided into three lots of 200 fruits each. The first lot was treated with 6% wax emulsion, i.e. 6 parts of emulsion base dissolved in 100 part of water, the second lot was treated with 3% wax emulsion and the third lot was kept as control. The wax treatment consisted of dipping the fruits in the emulsion for ca. 1 min followed by airdrying at ambient condition. Each of these lots was further subdivided into two. One lot being kept at  $50-55^{\circ}F$  and R.H.  $52.90^{\circ}$ , and the other at  $63 \pm 1^{\circ}F$  and R.H.  $90^{\circ}$ .

The weight of each lot of fruits was noted at weekly intervals and the percentage loss of moisture calculated. The juice of maltas from each lot was then expressed in an electrically operated juice-expressing machine and analysed for brix, total sugars, reducing sugars and acidity (expressed as citric acid), according to AOAC/methods<sup>6</sup> and vitamin C according to Barkat *et al.*<sup>7</sup>. The values for acidity and vitamin C content were also corrected for changes in brix due to storage according to the following formula:

Corrected value=  $V \times \text{initial brix}$ 

#### observed brix

where V stands for observed values of acidity i.e. mg of citric or mg of vitamin C per 100 ml juice.

Respiration rate was measured by continuous current method, the  $CO_2$  evolved was absorbed in KOH which was subsequently titrated against standard H<sub>2</sub>SO<sub>4</sub>. The results were expressed as mg CO<sub>2</sub> evolved per kg of fruits/hr.

Spoilage Due to Mould Growth.—The total number of fruits spoiled as a result of mould growth in all the lots was noted after the end of the experimental period of 5 weeks.

In another experiment, properly washed oranges, 450 in all, were divided into 2 lots of 225 oranges each. Fruits of one lot were deliberately damaged by rubbing on abrassive stone peeler. Fruits of both the damaged and undamaged lots were then inoculated with the mould spores found responsible to cause spoilage. These moulds, which formed green, bluish-green and black conidia were, in fact, isolated from spoiled/infected fruits. The green and bluish-green moulds belonged to Penicillium and the black was a species of Alternaria. For inoculation the fruits were dipped in the suspension of the spores in water. The inoculated fruits were air-dried and each lot was further divided into lots of 57 fruits each which were then separately treated (Chart I)

The fruits of the different lots were then stored at room temperature  $(60 \pm 5^{\circ}F)$  and at ca. 90% R.H.

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Lot 1.	Damaged fruits	No treatment (control).
,, 2.	,, ,,	Treated with 6% emulsion.
,, 3.	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	Treated with 3% emulsion.
,, 4.	Undamaged	No treatment (control).
,, ).	"" "	Treated with 6% emulsion.
,, 0.	,, ,,	Treated with 3% emuision.

Chart 1.

Organoleptic Evaluation.—The juice expressed from one dozen fruits from each lot was subject to to organoleptic evaluation by a taste panel selected from the staff of PCSIR Laboratories, Lahore.

## **Results and Discussion**

Loss of Moisture.—It will be observed from Table 1 that the loss of moisture in the treated fruits was much less than that in the control. Among the treated fruits the loss was more with 3%wax emulsion than those treated with 6% wax emulsion. It was also observed that fruits keept under humid conditions (R.H. 90%) even at a higher temperature (63°F) retained greater amounts of moisture than those kept under less humid conditions (55°F; R.H. 52–90 R.H.).

Brix, Sugar Content and Acidity.—Table 2-4, show that brix, total sugars and reducing sugars of juice increased with the passage of time but there was greater increase in the control than in the treated lots. The juice of the control lot had progressively less acidity than those of the treated lots. This is more clearly observed if the corrected figures for acidity shown in parenthsis in Table 5 are examined. Since increase in brix and sugar contents of fruit juice is associated with the loss of moisture and decrease in acidity is due to physiological changes as a result of ripening on storage, the delaying effect of wax treatment on the ripening process has been clearly demonstrated.

When comparing the effect of two treatments, i.e. with 3% and 6% wax emulsions, it was observed that the latter was more effective in checktng the build-up of concentration of sugars than the former under both the environmental conditions (i.e. at 50-55°F, R.H. 50-90% and  $63\pm$ °F, R.H. 90%).

Tables 2–5 show that there was no appreciable difference between the brix and sugar contents and acidities of fruit juices kept under the two different environmental conditions. It is also clear that high temperature accompanied by high humidity did not render the emulsion ineffective in extending the storage life.

Vitamin C.—Some loss of vitamin C invariably occurs on storage of fruits. It will be seen from Table 6 that although there was progressive decrease of vitamin C content of fruit juice in all the six lots, but the loss of vitamin C was more in the control than in the treated lots under both the environmental conditions. There appear to be no appreciable difference in checking the loss of vitamin C by applying 3 or 6% wax emulsion and the extent of losses was the same under both the environmental conditions.

TABLE I.—CUMULATIVE % LOSS OF MOISTURE IN VARIOUS LOTS OF ORANGES STORED UNDER DIFFERENT ENVIRONMENTAL CONDITIONS.

Treatment	1st week		2nd week 3rd week			week	4th v	week	5th week	
Treatment	A	В	A	В	A	В	A	В	A	В
Control	4.6	10.0	8.5	14	12.6	18.8	18.0	24.3	25.8	32.8
Wax emulsion $\binom{0}{0}$ Wax emulsion $\binom{0}{0}$	2.4 2.7	$3.5 \\ 4.2$	$\begin{array}{c} 4\cdot 4\\ 4\cdot 7\end{array}$	5.6 6.5	$5 \cdot 4 \\ 7 \cdot 5$	8.0 10.7	7.9 10.0	10.4 13.1	11.8 14.5	14.2 16.7

A=Lot of oranges kept at  $63 \pm 1^{\circ}$ F and R.H. 90%.

B=Lot of oranges kept at 50-55°F and R.H. 50-90%.

TABLE 2.—CHANGES IN BRIX OF JUICES OF VARIOUS LOTS OF ORANGES STORED UNDER DIFFERENT ENVIRONMENTAL CONDITIONS

Treatment	Initial reading		1st week		2nd week		3rd week		4th week		5th week	
	A	В	A	В	A	В	A	В	A	B	A	В
Control Wax emulsion (%) Wax emulsion (%)	6.0 6.0 6.0	6.0 6.0 6.0	6.0 6.0 6.0	6.5 6.2 6.2	$6.5 \\ 6.5 \\ 6.5 \\ 6.5$	7.0 6.2 6.5	$7.5 \\ 6.5 \\ 7.0$	7.5 7.0 7.0	7.5 7.0 7.0	8.0 7.0 7.0	8.0 7.0 7.0	8.5 7.0 7.0

A=Lot of oranges kept at 63±1°F and R.H. 90%.

B=Lot oforanges kept at 50-55°F and R.H. 50-90%.

T	Initial		Ist week		2nd week		3rd week		4th week		5th week	
Ireatment	A	В	A	В	A	в	А	в	A	в	A	В
Control Wax emulsion (6%) Wax emulsion (3%)	$5.7 \\ 5.7 \\ 5.7 \\ 5.7 $	$5 \cdot 7$ $5 \cdot 7$ $5 \cdot 7$	6.2 6.2 6.4	6.2 6.1 6.6	6.5 6.2 6.2	$   \begin{array}{r}     6.8 \\     6.3 \\     6.3   \end{array} $	7.7 6.4 6.5	7.7 6.5 6.5	7.9 6.8 6.6	$7.5 \\ 6.5 \\ 6.8$	8.0 6.5 6.8	8.0 6.5 6.6

TABLE 3.—CHANGES IN SUGAR CONTENT (g/100 ml) OF JUICES OF VARIOUS LOTS OF ORANGES STORED UNDER DIFFERENT ENVIRONMENTAL CONDITIONS.

A=Lot of oranges kept at  $63\pm1^{\circ}F$  and R.H. 90%. B=Lot of oranges kept at 50-55°F and R.H. 50-90%.

TABLE 4.—CHANGES IN REDUCING SUGAR CONTENT (g/100 ml) OF JUICES OF VARIOUS LOTS OF ORANGES, STORED UNDER DIFFERENT ENVIRONMENTMENTAL CONDITIONS.

Tractoret	Initial		ıst w	1st week		2nd week		3rd week		4th week		5th week	
Ireatment	A	в	А	в	Α	В	A	В	A	В	A	В	
Control Wax emulsion (6%) Wax emulsion (3%)	2.5 2.5 2.5	2.5 2.5 2.5	2.8 3.0 3.0	3.1 3.0 3.1	3.0 2.8 2.2	3·3 2.7 2.9	$3 \cdot 4 \\ 3 \cdot 3 \\ 3 \cdot 9$	$3 \cdot 4 \\ 3 \cdot 3 \\ 3 \cdot 4$	4.0 3.0 3.2	$3.6 \\ 3.0 \\ 3.3$	$4.0 \\ 3.3 \\ 3.5$	$4 \cdot 3$ $3 \cdot 3$ $3 \cdot 5$	

A=Lot of oranges kept at  $63\pm1^\circ$ F and R.H. 90%. B=Lot of oranges kept at 50-55°F and R.H. 50-90%.

TABLE 5.—CHANGES IN ACIDITY (g OF CITRIC ACID/100 ml) OF ORANGE JUICES OF VARIOUS LOTS STORED UNDER DIFFERENT ENVIRONMENTAL CONDITIONS.

	Initial 1st			week 2 week		3rd week		4th week		5th week		
Treatment	A	В	A	В	A	В	Ā	В	A	в	A	В
Control	0.67	0.67	0.51	0.50 (0.47)	0.51 (0.47)	0.45 (0.39)	0.44 (0.33)	0.46 (0.37)	0.46 (0.36)	0.46 (0.34)	0.45 (0.33)	0.42 (0.31)
Wax emulsion 6%	0.67	0.67	0.48	0.50 (0.50)	0.48 (0.44)	0.45 (0.42)	0.48 (0.44)	0.47 (0.40)	0.41 (0.36)	0.43 (0.37)	0.45 (0.38)	0.47 (0.40)
Wax emulsion 3%	0.67	0.67	0.47	0.51 (0.50)	0.46 (0.42)	0.43 (0.40)	0.46 (0.40)	0.46 (0.40)	0.41 (0.36)	0.47 (0.40)	0.48 (0.41)	0.48 (0.41)

A=Lot of oranges kept at  $63\pm1^{\circ}F$  and R.H. 90%. B=Lot of oranges kept at 50--55°F and R.H. 50-90%.

(Figures in paranthesis show the corrected value for changes in brix.)

# TABLE 6.—CHANGES IN VITAMIN C CONTENT (g/100 ml) OF JUICES OF VARIOUS LOTS OF ORANGES STORED UNDER DIFFERENT ENVIRONMENTAL CONDITIONS.

Treatment	Initial		1st week		2nd	2nd week		3rd week		4th week		eek
	A	В	A	В	A	В	A	В	A	в	A	В
Control	54	54	52	50 (46)	50 (46)	55 (47)	50 (40)	50 (40)	46 (37)	46 (34)	46 (34)	40 (30)
Wax emulsion $6\%$	54	- 54	50	50 (48)	52 (48)	50 (48)	48 (44)	48 (41)	48 (42)	48 (41)	48 (41)	50 (43)
Wax emulsion 3%	54	54	20	52 (50)	56 (52)	53 (48)	50 (43)	56 (48)	48 (41)	48 (41)	48 (41)	48 (41)

A=Lot of oranges kept at  $63\pm1^{\circ}$ F and R.H. 90%

B=Lot of oranges kept at 50-55°F and R.H. 50-90%.

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TABLE 7.—TASTE PANEL SCORES OF FRUIT FOR DIFFERENT LOTS OF ORANGES. AVERAGE OF 7 JUDGES

Treatment	Appear- ance	Taste	Flavou	
Control	6.0	5.6	5.4	The difference were
Fruitex 3%	6.1	5.6	5.3	ficant between various
Fruitex 6%	6.0	5.6	5.3	ferent lots.

Respiration Rate.—The respiration rates measured at regular intervals up to 4 weeks are graphically shown in Fig. 1. It is evident from the curves that the rate of  $CO_2$  liberation was much less in the treated fruits as compared with the control. Among the treated fruits the rate was less in fruits treated with higher concentration of wax emulsion. The average respiration rates during the entire period under study in the untreated (control) and in the 6 and 3% emulsion treated fruits were respectively 21.1, 8.8 and 12.4 mg  $CO_2/kg/hr$ .

Spoilage Due to Mould.—Spoilage due to mould growth during storage for 8 weeks are given in Fig. 2. Among the damaged fruit lots, whereas there was almost 100% wastage due to moulds of the untreated fruits within 2 weeks, only 46 and 58% of fruits treated with 6 and 3% wax emulsion, respectively, were spoiled during the same period. However, 75 and 84% of 6 and 3% wax-treated fruits were spoiled within 8 weeks stored at 63°F and R.H. 90%.

Among the undamaged fruit the rate of spoilage, as expected, was lower than the damaged fruits. Spoilage of the 6% wax-treated fruits was always less than either of the untreated or 3% wax-treated fruits, at any stage during the storage period up to 8 weeks.

The results of the effect of the wax treatment of the fruits particularly the damaged fruits, clearly demonstrate the fungicidal value of the wax emulsion. Both the growth and conidia formation of the causative moulds were checked by the treatment.

The relatively high rate of spoilage of fruits under the condition should not be taken seriously as the fruits for the experiment were exposed to extreme conditions favouring prompt mould growth on the fruits and their subsequent spoilage. However, it would be quite desirable, if the wax emulsion has increased fungicidal value and is more effective and versatile under all sorts of conditions. Additions of certain metallic salts have been found to increase the fungicidal value of the emulsion. This is being studied and will be reported in future communication.

Organoleptic Evaluation of Fruit Juices.—The results of organoleptic tests made on different lots of fruits are shown in Table 7. It will be observed



Fig. 1.-Respiration sates of variously treated oranges.



Fig. 2.—Spoilage of variously treated oranges.

that there was no statistically significant difference between appearance, taste and flavour of differently treated lots of fruits.

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# References

- I. Data provided by the Government of Pakistan, Agricultural Department (1967 - 68).
- 2. E. J. Miller, Mich. State Univ. Agr. Exp. Stat. Tech. Bull. No. 218 (1950).
- 3. R. Griffith, U.S. Pat. 2,288, 351; Chem. Abstr., 37, 196 (1943).
- J. C. Towner., Aust. Pat., 116,036, Nov. 5, 4. 1942; Chem. Abstr., 40, 753 (1946).
- 5. Process for the Production of Fungicidal Wax Emulsion, Shaukat Ali, M. Aslam, Ali Ehsan, Pakistan Patent No. 121, 176 (1971).
- Methods of Analysis (George Banta Publishing 6. Co., Wisconsin, 1960), 9th edition, p. 271. 7. M.Z. Barkat, M.F., El-Wohob and M.M.
- El-Sadar, Anal. Chem., 27, 536 (1955).