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著者	ASO Kiyoshi, NAKAYAMA Teizo, SATO Akio
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STUDIES ON THE UTILIZATION OF GRAPE
PART V. COLOR CHANGE OF CONCORD GRAPE JUICE BY VARIOUS TREATMENTS AND STORAGE*

By

Kiyoshi ASO, Teizo NAKAYAMA and Akio SATO

Department of Agricultural Chemistry, Faculty of Agriculture, Tohoku University, Sendai, Japan

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Concord grape is most valued as the raw material for grape juice, one of the most popular fruit beverages. The color of the matured Concord grape juice is a fairly intense reddish purple. This is ranked as an important factor for judging the quality of the juice as well as its taste and aroma. The characteristic color of Concord grapes has been attributed principally to the water-soluble anthocyanin pigments. Recently, Sastry and Tischer (1) pointed out the presence of chlorophyll, water-soluble yellow pigments, and carotins in addition to the anthocyanins.

In studying the visual color changes occurring in Concord grape juice during storage, Pederson and Tressler (2) noted that the color changed from reddish purple to a brick red and then to a dull brown simultaneously with clouding and sedimentation. Nebesky *et al* (3), studying the effects of various factors on the stability of the color in a variety of fruit juices during storage, observed that cherry, grape, and tomato juices were much more stable and exhibited less color deterioration than strawberry, raspberry, and currant juices. Working with purified pigment solutions from strawberries and currants, they observed that high storage temperatures and the presence of oxygen accelerated color deterioration with both pigments, while a low storage temperature preserved the color. Pederson (4) described in "Fruit and Vegetable Juice Production" that the deterioration of the color is caused by the presence of oxygen of the air and by prolonged heating. Sastry and Tischer (5) stated that the period of storage exerted the maximum influence on the deterioration of the anthocyanin pigments of Concord grape juice, and both of the mono- and diglucosides of the anthocyanidin decreased on storage. They also stated that the decrease in the pigment concentration on processing and storage was more striking in the case of anthocyanin chloride solutions than in grape juice samples.

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The present work was made to study the effects of various treatments in the preparation of grape juice on the color changes. The color changes of grape juice by pasteurization and storage under controlled experimental conditions were also investigated.

Experimental

Concord grapes were washed, stemmed, crushed, heated up to 60°C, and kept at this temperature for 15 minutes. The cooked mass was pressed by the laboratory press and centrifuged. And then, various treatments such as carbonation, argol-removal, addition of sugar or of tartaric acid, and cation-exchange were tried on the resulting juices. For carbonation, carbon dioxide was introduced into the juice up to 760 mm Hg after the headspace of the flask containing juice was reduced to 300 mm Hg. This process was repeated four times and finally the introduction of carbon dioxide was continued for ten minutes at 760 mm Hg. Amberlite IR-120 was employed for cation-exchange. The removal of argols was made under the condition that the juice was kept at 0°C for 60 hours. All the juice samples were filled into the 180 ml colorless glass bottles and pasteurized at 75°C for 30 minutes.

In addition, a portion of the grapes was cold-pressed without stemming, and the residual components in pomace were extracted with hot water. After the cold-pressed juice and the pomace extract were separately concentrated below 30°C, the mixture of both was used as the concentrated juice sample. A portion of the concentrated juice was blended with the 1st, 2nd, and 3rd essences recovered by the essence equipment, as reported previously (6). To this blended juice, water and sucrose were added so that one volume of the original juice was diluted to three volumes and the total sugar content was adjusted to approximately 12 per cent. Thus, the resulting juice was used as the commercial juice sample. The carbonated commercial juice was prepared by the addition of water saturated with carbon dioxide instead of water, followed by introducing more carbon dioxide into the bottled juice solution for ten minutes. These samples were also in the 180 ml colorless glass bottles, and pasteurization was conducted at 75°C for 20 minutes. The pasteurized juice samples were stored for about eight months at three different temperatures; 30°C, room temperature, and 0°C The effect of light was also examined with the samples stored at room temperature.

The color measurement of the juice samples was made on the principle of the previous method (6). The juice for color measurement was adjusted to the same acidity as the original one with a mixture of tartaric and malic acids (1:1) and clarified with ethanol. The optical densities of the clear solutions were measured with the Hitachi spectrophotometer EPO-B in the entire visible range. The intensity and character of color were represented with Total DC and color distribution ratio, respectively.

Results and Discussion

Total DC and color distribution ratio of the original juices, the concentrated juice, and the commercial juices are included in Table 1. Those of the juice samples prepared by various treatments from the original juices are summarized in Table 2. From the data it is known that there was an increase in Total DC with the addition of sucrose or tartaric acids while a decrease in Total DC occurred with carbonation, removal of argols, and cation-exchange. The change of Total DC by carbonation is negligible, but the decrease by cation-exchange and the increase by addition of tartaric acid, especially 2 to 3 per cent, gave a fairly serious influence on the color. The decrease of Total DC by removal of argols might be due to the precipitation of pigments which occurred during storage at low temperature to remove the argols, but the color change of juice was not so noticeable. The increase of Total DC by the addition of sucrose, which was relatively small, was thought to be based on lower pH than the original one. Namely, although the titratable acidity was adjusted to the same as the original with the malic and tartaric acid mixture (1:1), any change of pH is possible since the ratio between malic and tartaric acid in the original juice is not accurately 1:1 and the former is slightly greater than the latter. It can be also explained from the drop of pH that the addition of tartaric acid brought about an increase of Total DC. The ion-exchange treatment caused the most important color change because the greater part of the pigments was adsorbed to cation-exchange resins.

The data also indicate that color distribution ratios of all other samples than cation-exchanged ones are similar to those of the original juices. With the cation-exchanged sample, the portion of reddish purple or red was remarkably decreased and contrarily the orange portion was strikingly increased.

Total DC and color distribution ratio of samples after pasteurization are shown in Table 3. The values presented for Total DC indicate that the decrease

Table 1. Total DC and color distribution ratio of the original juices, concentrated juice and commercial juices

		Total DC	Color distribution ratio filter 43	47	50	53	55	57	61	66
Original juice	I	21.512	10.90	13.39	19.34	22.39	19.52	10.00	3.01	1.49
	II	23.728	10.42	12.98	19.49	22.86	19.79	10.05	2.97	1.45
	III	21.968	10.74	12.82	20.03	21.85	20.47	10.01	2.91	1.17
Concentrated juice		64.032	12.37	13.42	18.70	21.93	19.12	9.78	3.07	1.61
Commercial juice I		3.891	15.20	14.65	17.50	18.97	16.81	9.79	4.47	2.39
Commercial juice II		3.732	15.59	14.71	17.97	19.12	17.08	9.89	4.33	2.45

Commercial juice I = Non-treated juice, Commercial juice II = Carbonated juice

Table 2. Total DC and color distribution ratio of the original juices after treatments

Original juice	Sample No.	Treatment	Total DC	Change of Total DC, %	Color distribution ratio filter 43	47	50	53	55	57	61	66
I	1	Non-treatment	21.512		10.90	13.39	19.34	22.39	19.52	10.00	3.01	1.49
	2	Carbonation	21.064	−2.1	11.01	13.29	19.29	22.22	19.75	9.87	2.96	1.60
II	3	Non-treatment	23.728		10.42	12.98	19.49	22.86	19.79	10.05	2.97	1.45
	4	Argol-removal	20.938	−11.8	11.10	13.07	19.28	22.43	19.89	9.60	3.07	1.57
	5	Sucrose-addition	24.915	+ 5.0	10.31	13.03	19.23	22.91	20.42	9.99	2.76	1.34
	6	Cation-exchange	6.471	−72.7	14.76	14.66	18.32	19.29	17.96	9.82	3.61	1.58
III	7	Tartaric acid-addition, 1%	24.040	+ 9.4	9.98	12.95	19.80	22.63	20.77	9.98	2.70	1.21
	8	Tartaric acid-addition, 2%	27.205	+23.4	10.79	12.90	19.50	23.10	20.40	9.60	2.61	1.11
	9	Tartaric acid-addition, 3%	27.761	+25.1	10.75	12.76	19.44	23.45	20.42	9.71	2.46	0.95

Table 3. Total DC and color distribution ratio of the juice samples after pasteurization

		Sample No.	Treatment	Total DC	Decrease of Total DC, %	Color distribution ratio filter 43	47	50	53	55	57	61	66
Original juice	I	1	Non-treatment	20.104	6.6	11.74	13.57	18.98	22.09	18.78	10.03	3.22	1.09
		2	Carbonation	19.192	8.9	12.13	13.30	18.59	21.88	19.09	10.00	3.33	1.67
	II	3	Non-treatment	22.104	6.8	11.22	13.43	19.00	22.62	19.62	9.70	3.00	1.41
		4	Argol-removal	18.276	11.2	12.17	13.53	21.87	20.74	17.58	9.33	3.16	1.62
		5	Sucrose-addition	22.534	9.5	12.01	13.54	18.73	22.05	18.69	9.96	3.36	1.60
		6	Cation-exchange	6.603	1.8	14.99	15.00	18.24	19.99	17.24	9.50	3.40	1.60
	III	7	Tartaric acid-addition, 1%	21.816	9.2	11.18	12.35	19.14	22.37	19.81	9.83	2.93	1.39
		8	Tartaric acid-addition, 2%	23.525	13.5	10.75	12.90	19.35	23.17	19.49	9.99	2.95	1.39
		9	Tartaric acid-addition, 3%	23.888	13.1	10.62	13.42	19.49	23.11	19.56	9.97	2.73	1.17
Concentrated juice				63.216	1.3	12.89	13.78	18.30	21.26	18.68	9.82	3.21	1.51
Commercial juice I				3.660	5.9	16.23	15.00	17.46	18.77	16.39	9.59	4.10	2.46
Commercial juice II				3.699	0.9	15.90	15.25	17.68	18.65	16.30	9.65	4.14	2.43

Commercial juice I = Non-treated juice, Commercial juice II = Carbonated juice of Total DC by pasteurization is several to 14 per cent. In comparing the samples prepared by various treatments from the original juices with the concentrated and commercial juice samples, the decrease per cent of the formers were found to be generally higher than one of the latters. This may be due to the longer pasteurization time of the formers than of the latters. But, only the cation-exchanged sample increased slightly in Total DC by pasteurization. It is not apparent why such a phenomenon happened.

Before pasteurization, the color distribution ratios (optical density per cent of every filter) of the juice samples determined over a range of 420 to 670 mμ using a Hitachi spectrophotometer EPO-B exhibited the maxima at filter 53 for all samples. By pasteurization, generally, the optical densities at filter 53 were lowered and those at filter 43 raised, which suggested that the anthocyanin pigments were destroyed by heating and its degradation products possessed higher absorption values around 400 mμ. Nevertheless, the optical densities at filter 53 after pasteurization still showed the maxima in a range of 420 to 670 mμ with the exception of the argol-removed sample having the maxiamum absorption at filter 50 after pasteurization.

The color changes of juice samples resulting from storage for about eight months under various conditions are presented in Table 4. Total DC changes of the non-treated and carbonated juice samples prepared from the original juice I during storage are shown in Figuer 1, those of the argol-removed and sugar-added juice samples in Figure 2, and the cation-exchanged and tartaric acid-added juice samples are given in Figure 3. The Total DC changes of the concentrated and commercial juice samples during storage are showh in Figure 4.

From these results it is known that the Total DC decreased during storage in every case, and the storage at 30°C caused the greatest decrease of Total DC and the storage at 0°C the least. The results also indicate that the difference between the samples stored in the dark and those exposed to light at room temperature was not clear. As shown in Figure 1, the carbonated sample gave results similar to the non-treated sample. The concentrated juice sample also showed similar results (Figure 4). Figure 3 shows that the cation-exchanged juice is very small in the decrease of Total DC during storage. This might be due to the small amount of pigments to be deteriorated because most of the pigments were already adsorbed to the resins. The argol-removed and sugar-added juice samples are, as known from Figure 2, smaller in the decrease of Total DC during storage than the non-treated original juice sample, but only from this experiment it is difficult to conclude that the treatment of argol-removal or sugar-addition protects the Concord grape pigments from deterioration. With the tartaric acid added juice sample, the decrease of Total DC during storage showed no definite relation with the amount of tartaric acid added. However, the data shown in these Figures and Table 4 pointed out that

Table 4. Total DC and color distribution ratio of the juice samples after storage

Juice sample			Condition of storage	Total DC	Decrease of Total DC, %	Color distribution ratio filter 43	47	50	53	55	57	61	66
Original juice	I	Sample No. 1	30°C, D	10.368	48.4	23.53	19.21	17.36	12.73	11.42	8.02	4.86	2.85
			R, L	12.168	39.5	18.54	17.55	18.54	15.98	13.48	8.88	4.73	2.30
			R, D	11.600	42.3	19.17	17.10	17.86	16.90	14.00	8.97	3.93	2.07
			0°C, D	13.784	31.4	14.57	15.61	19.33	20.95	16.60	8.88	2.84	1.22
		2	30°C, D	9.512	50.4	22.88	18.76	17.83	13.62	11.10	8.41	4.71	2.69
			R, L	11.848	38.3	18.56	17.22	17.69	16.61	14.25	9.25	4.25	2.16
			R, D	11.368	40.8	19.14	17.66	17.80	16.75	13.79	8.94	4.01	1.90
			0°C, D	13.112	31.7	14.95	15.80	19.34	20.68	16.17	8.91	2.93	1.22
	II	3	R, L	11.768	46.8	19.17	17.13	17.88	16.66	13.87	9.31	4.08	1.90
			R, D	12.160	45.0	19.01	17.30	17.89	16.84	14.14	9.01	3.95	1.84
		4	R, L	11.379	37.7	17.81	16.44	17.81	16.51	15.35	9.63	4.31	2.32
			R, D	11.412	37.6	18.12	16.61	17.69	16.46	15.02	9.31	4.48	2.31
		5	R, L	13.146	41.7	17.96	16.24	17.14	16.32	14.74	9.81	4.87	2.92
			R, D	13.274	41.1	18.16	16.38	17.05	16.68	14.91	9.49	4.60	2.74
		6	R, L	5.504	16.6	23.32	19.90	17.79	13.61	11.93	7.49	3.66	2.22
			R, D	5.732	13.2	22.34	19.23	17.79	14.51	12.55	7.77	3.57	2.25
	III	7	R, L	14.080	35.5	15.91	15.80	17.56	18.18	16.42	9.77	3.98	2.39
			R, D	14.048	35.6	15.66	15.43	18.22	18.68	16.23	9.45	4.04	2.28
		8	R, L	14.476	38.5	16.01	15.84	18.60	18.71	16.80	9.13	3.55	1.35
			R, D	14.372	38.9	15.32	15.95	17.82	19.01	17.25	9.76	3.52	1.36
		9	R, L	14.923	37.5	14.36	16.18	18.33	18.88	17.39	9.88	3.59	1.38
			R, D	15.022	37.1	14.92	15.47	17.94	19.64	17.83	9.38	3.46	1.37
Concentrated juice			30°C, D	30.768	51.3	24.10	18.72	16.22	12.87	11.31	8.35	5.07	3.35
			R, L	34.392	45.6	18.98	16.05	17.10	16.75	15.14	9.49	4.12	2.37
			R, D	35.040	44.6	18.63	15.82	17.05	17.19	15.41	9.18	4.11	2.60
			0°C, D	46.800	26.0	14.82	14.87	18.92	20.67	17.38	9.13	2.92	1.28
Commercial juice I			R, L	3.111	15.0	23.34	19.58	17.36	13.60	12.34	7.81	3.76	2.22
			R, D	3.108	15.1	23.34	19.58	17.36	14.19	13.03	8.49	3.96	2.41
Commercial juice II			R, L	3.021	18.3	22.84	18.97	17.38	14.10	12.41	8.24	3.77	2.28
			R, D	2.868	22.5	21.44	18.72	17.78	14.64	13.28	8.47	3.56	2.09

Commercial juice I = Non-treated juice, Commercial juice II = Carbonated juice
R = Room temperature, D = Dark, L = Light

the period of storage exerted the maximum influence on the deterioration of Concord grape color and suggested that the rate of deterioration of color is proportional to the storage temperature.

The curves of color distribution ratios of the juice samples before and after storage are shown in Figures 5–8. The carbonated juice samples showed the same curves as those of the non-treated samples and the argol-removed, sugar-added, and tartaric acid-added juice samples gave almost the same curves as those of the non-treated samples stored at room temperature. The results indicate that in all the samples there was a decrease of optical density at filter

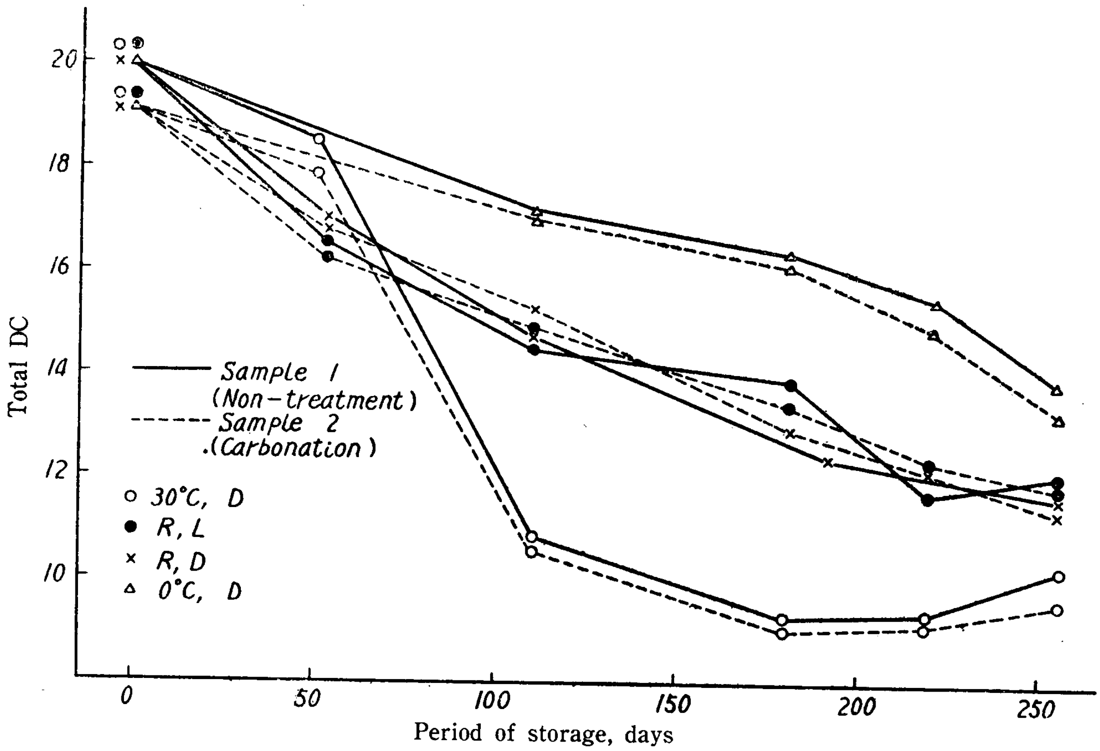

Fig. 1. Total DC changes of sample 1 and 2 during storage (D=Dark, L=Light, R=Room temperature)

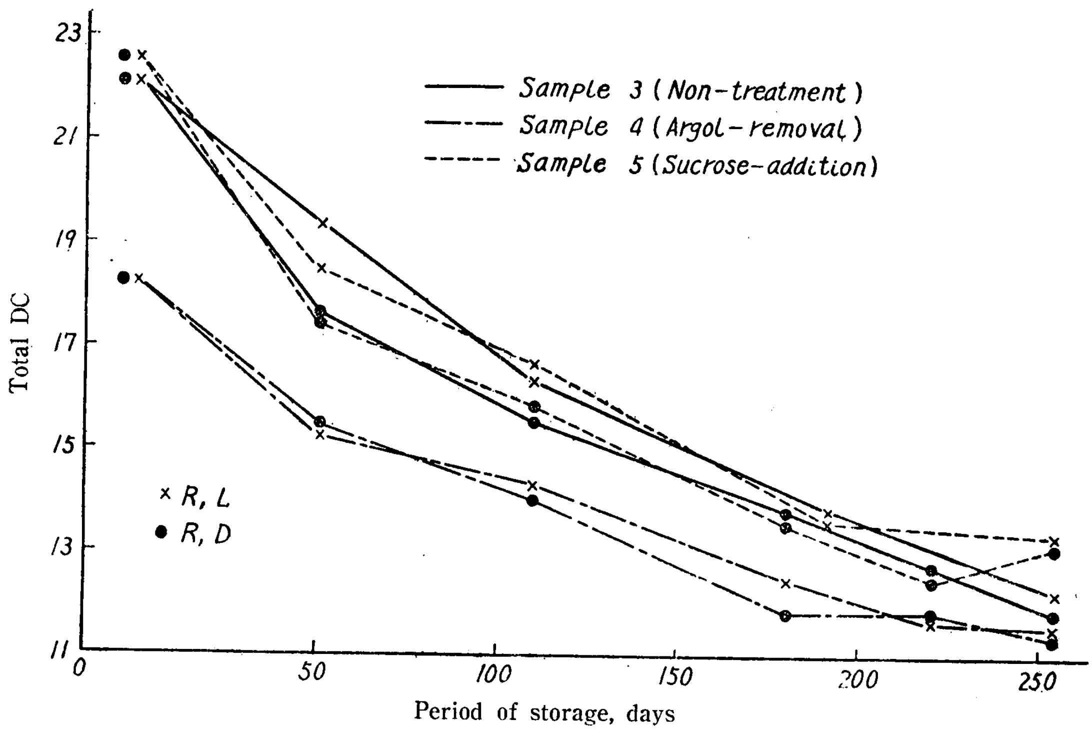

Fig 2. Total DC changes of samples 3, 4, and 5 during storage (D=Dark, L=Light, R=Room temperature)

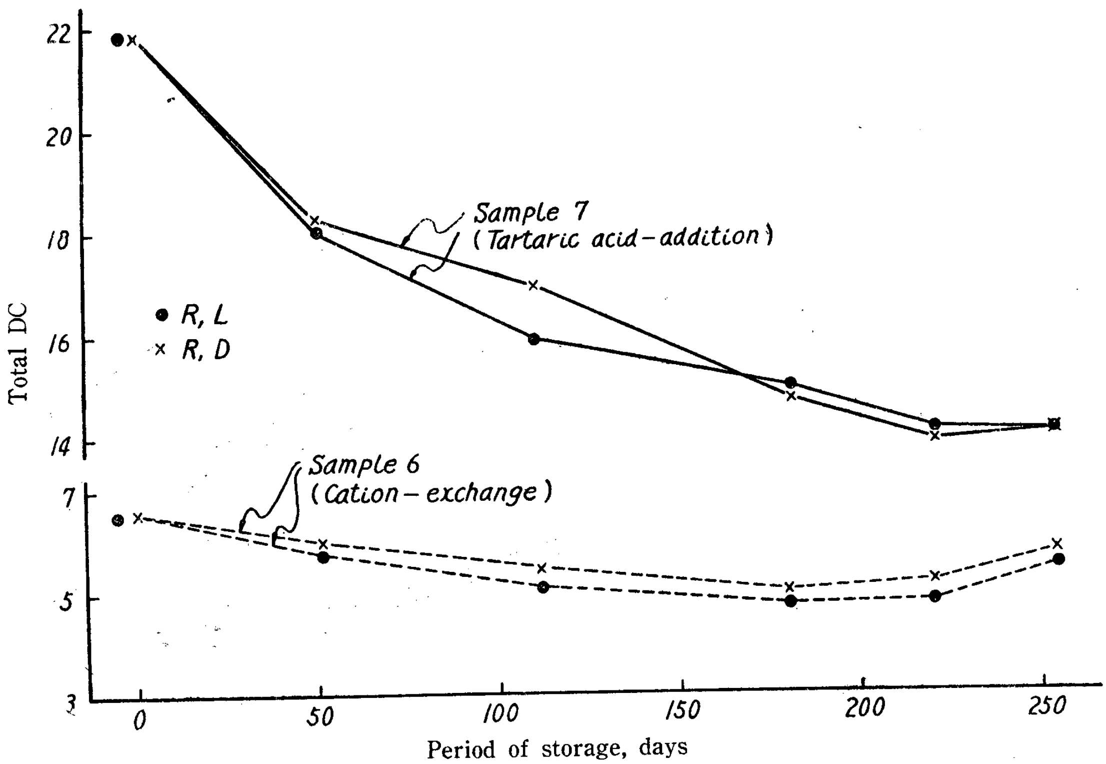

Fig 3. Total DC changes of sample 6 and 7 during storage
(D=Dark, L=Light, R=Room temperature)

53 and an increase of optical density at filter 43 and 47 under the storage conditions in this experiment, and the storage temperature brought about the maximum influence on the change of color character. It was observed from the point of visual change of color that the color of the juices stored at 0°C hardly changed, the juices at the room temperature turned into brick red and the juice at 30°C into dull brown with brown sediment. Various treatments such as carbonation, addition of sugar or of tartaric acid, removal of argols, and cation-exchange provided no accelerative or preventive effect on the degradation of the Concord grape color during storage. The effect of light was negligible on the deterioration of color in most cases, but the cation-exchanged juice and the commercial juice samples showed somewhat noticeable difference between the dark and the light. In the case of these samples, the light could penetrate more easily into the interior of the juice since the concentrations of color components and colloidal matters were reduced.

In conclusion, the color changes occurring in the Concord grape juice during storage are the decrease of Total DC (the decrease of color intensity) and the changes of color distribution ratio (the deterioration of color character). The changes by pasteurization also indicate the same tendency. The deterioration of color character results from the decrease of absorption at filter 53, namely the decrease of reddish purple, and simultaneously from the increase of absorption at filter 43, namely the increase of yellow. Sastry and Tischer (1, 5) reported that the characteristic color of Concord grape is attributed principally to the mono- and diglucosides of the anthocyanidin, which have the absorption maxima at 515 mμ in the visible range. They also described that by heating or other treatments the absorption maxima of these glucosides were shifted toward 400 mμ in the visible range. As above mentioned, our experimental results agree with theirs. In addition to the degradation of anthocyanin pigments, other changes such as a Maillard reaction, deterioration of pectic substances, of ascorbic acid or of polyphenolic substances may also have occurred in the grape juice. The absorption maxima can also be shifted by these browning reactions. It may illustrate the occurrences of these browning reactions in the grape juices that the Total DC curves during storage at 30°C showed a little increase in the later period of storage.

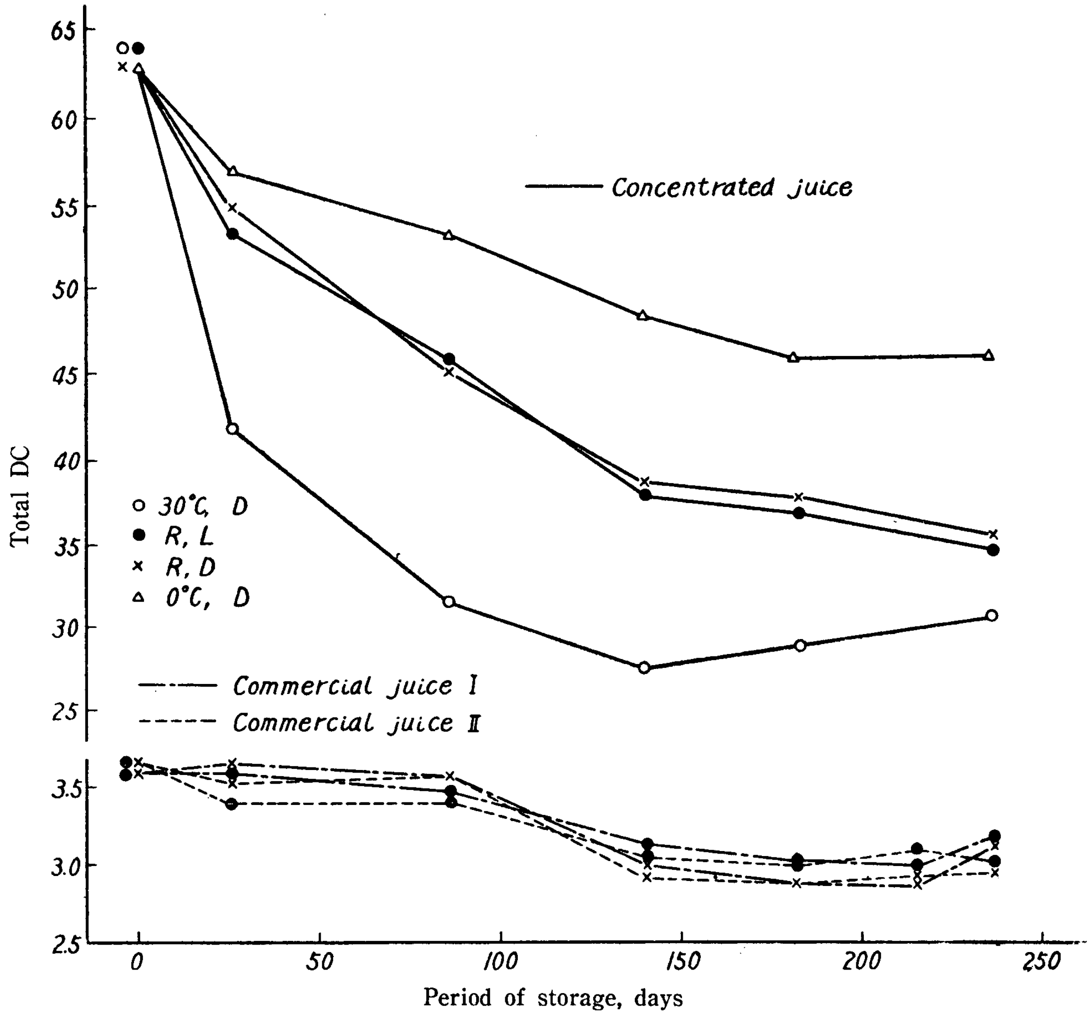

Fig. 4. Total DC changes of concentrated juice and commercial juice during storage (D=Dark, L=Light, R=Room temperature)

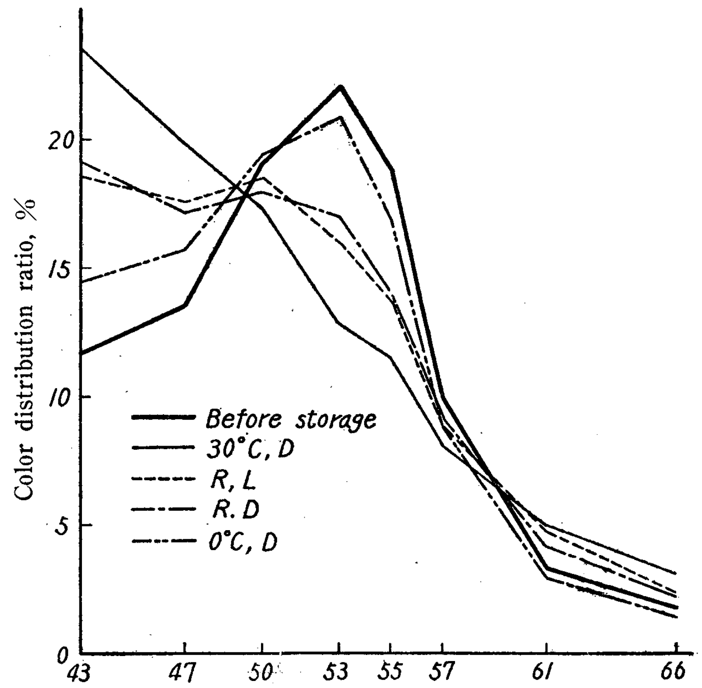

Fig 5. Color distribution ratio of sample 1 (non-treatment) after storage (254 days).
(D=Dark, L=Light, R=Room temperature)

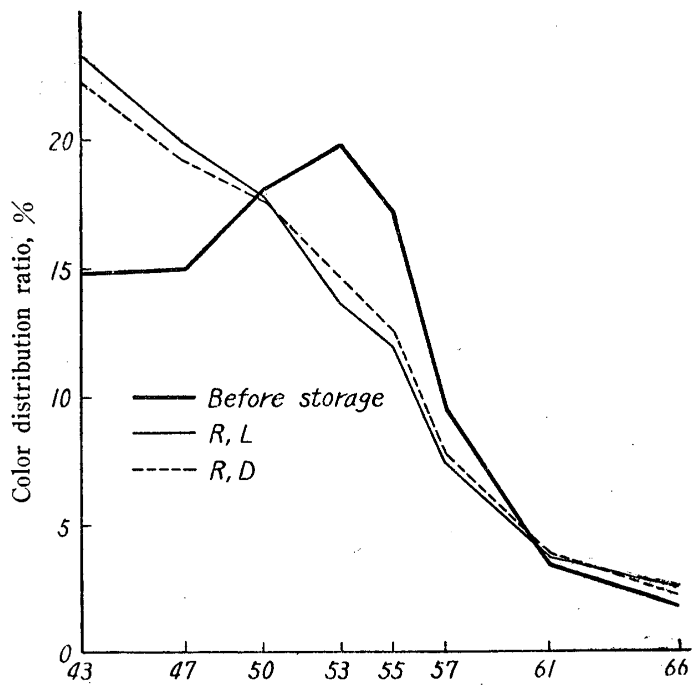

Fig. 6. Color distribution ratio of sample 6 (cation-exchange) after storage (255 days).
(D=Dark, L=Light, R=Room temperature)

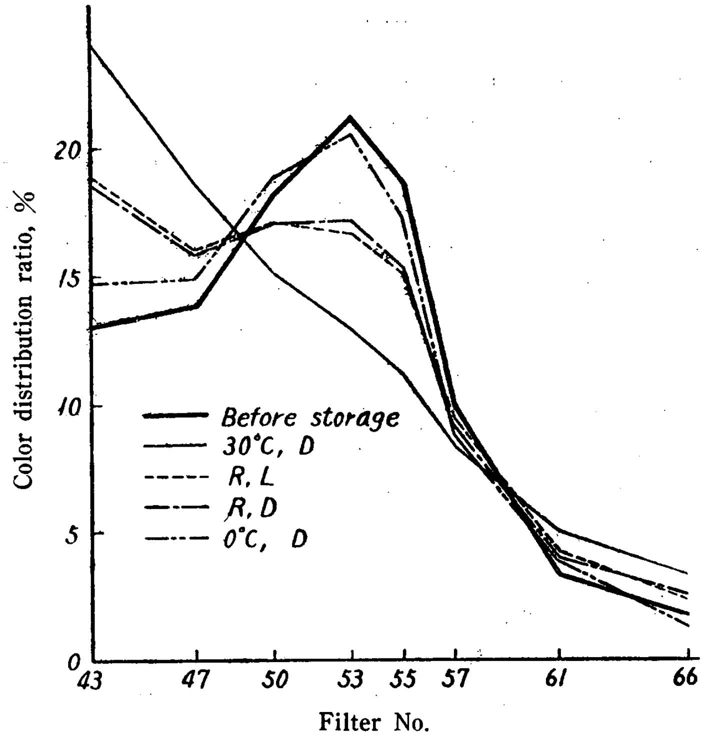

Fig. 7. Color distribution ratio of concentrated juice after storage (234 days).
(D=Dark, L=Light, R=Room temperature)

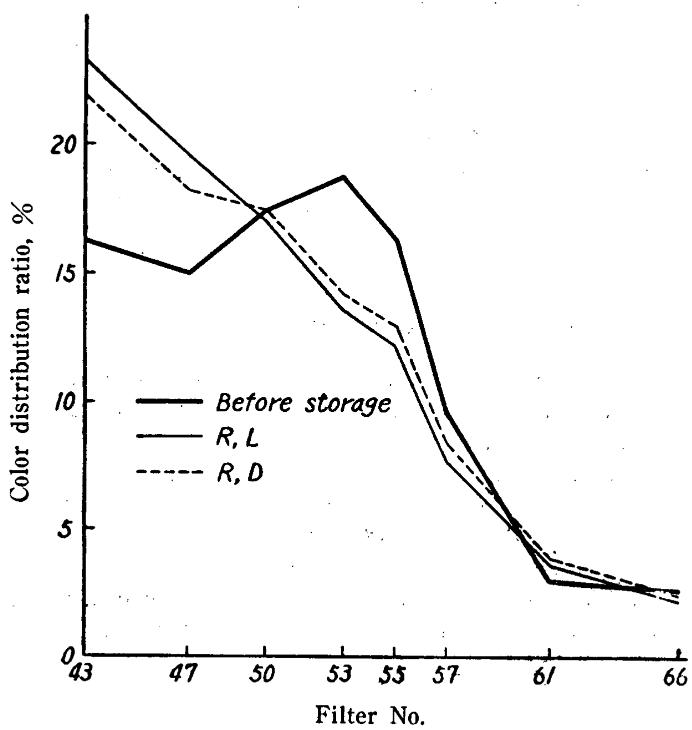

Fig. 8. Color distribution ratio of commercial juice after storage (234 days)
(D=Dark, L=Light, R=Room temperature)

Summary

Various treatments such as carbonation, argol-removal, sugar-addition or tartaric acid-addition gave no important influence on the color of Concord grape juice, but the effect of cation-exchange by Amberlite IR-120 was so important that the juice color changed to light orange because the greater part of the pigments were absorbed to resins. On storage as well as pasteurization, the color of Concord grape juice changed from reddish purple to a brick red and then to a dull brown simultaneously with the decrease in the color intensity. By investigating the effects of various factors on the color during storage under controlled experimental conditons, it was observed that the period of storage and the storage temperature exerted the maximum influence on the deterioration of the Concord grape color. The treatments such as carbonation, argol-removal, sugar-addition, tartaric acid-addition or cation-exchange provided no accelerative or preventive effect on the deterioration of the Concord grape color during storage. The juice samples after storage at 30°C for six months showed a decrease of 50 per cent in the color intensity and the color character changed to a dull brown with simultaneou formation of clouding sediments in the solution, while the juice samples afters storage at 0°C for eight months retained 70 per cent of the initial color intensity and the change of color character was very small. The color changes occurring in the concentrated juice during storage were also similar to these results. The effect of light was only of minor importance in the deterioration of color of most juice samples, but the cation-exchanged juice and the commercial juice samples showed somewhat noticeable difference between the dark and the light. In the case of these samples, the light could penetrate more easily into the interior of the juice because the concentrations of color components and colloidal matters were reduced.

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