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# Investigation of characteristic colour stability of powdered orange

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#### Abstract

The paper presents stability investigations of characteristic colour of powdered orange used for juice production. The colour of packed products darkens during the 12-month storage. In the same period, changes of characteristic wavelength are cyclical. These changes depend on the kind of combined packaging material used for packing the investigated product, as well as on the applied packing conditions. The product colour change depends on the barrier characteristics of the packaging material and its permeability to water vapour and oxygen. The permeability of light has no major influence on colour change, while packing under vacuum has the greatest protective effect.  $\odot$  2000 Elsevier Science Ltd. All rights reserved.

# 1. Introduction

In time, the composition of every packed product changes and such changes are defined as "shelf-life". Changes are especially expressed in dehydrated powdered products due to the high concentration of components and large active surface of the powder. Other factors, such as storage time, light, moisture and oxidation also affect the changes of packed products. The specific colour of a particular product is one of the significant quality characteristics. Colour changes are connected with change of spectral properties of the product constituents.

Colour stability of a packed product depends on a great number of factors. A number of investigators have dealt with colour stability of products using model systems or finished products (Eichner, 1975; Granzer, 1982; Karel & Nickerson, 1964; Kreisman & Labuza, 1978; Labuza & Saltmarch, 1980; Rockland & Nishi, 1980; Schobinger & Dürr, 1980; Waletzko & Labuza, 1976).

The colour of a product is the visual effect of subjective sensation of the observer. Every coloured substance selectively absorbs the light of the visual part of the spectrum. When white sunlight shines upon a coloured molecule, a certain part of the light spectrum is absorbed while the other is reflected and creates the impression of colour in the observer's eye. The coloured light beam which reaches the eye, is in the range of wavelengths of dominant colours, and the intensities of particular wavelengths are variable. Pure colours, defined with only one wavelength, do not exist. Each colour is characterized by the sum of wavelengths of different intensities and can be presented as a dominant wavelength.

Brightness is an optical quality of colour and presents a measure of intensity of the colour sensation. Brightness means the average reflectance and is classified by the range in which white colour is the highest brightness and black colour the lowest; e.g. higher values of average reflectance represent higher brightness and lower values, darker colour, i.e. lower brightness. The increase of brightness defines the darkening of the product (Heredia & Guzman Chozas, 1992; Robertson, 1972).

The measurement of colour quality of powdered fruit is a big problem since light disperses due to the large active surface of the analyzed sample. The chosen method of measurement and following of characteristic colour quality is one of the bases of determination of average reflectance and dominant wavelength of a product.

Eagerman (1978), Huggart, Barron and Wenzel  $(1966)$  and Rummens  $(1970)$  discussed the effect of optical geometry on measured colour values of orange juice.

The changes of total colour quality of a product depend on its initial quality, packaging used, packing

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conditions, type and protecting properties of packaging material combination in relation to light, humidity and oxidation. In order to minimize the changes during storage, it is necessary to choose the appropriate combination of materials which will keep the quality unchanged for a longer period of time (Eichner & Karel, 1972; Gvozdenović et al., 1978, 1981, 1993; Hanlon, 1965; Heiss, 1980; Lucae, 1978; Varsanyi, 1985).

Permeability of light, molecules of gases from the air (oxygen, nitrogen and carbondioxide), as well as of water vapour, are the main characteristics defining the barrier properties of packaging materials. In order to maintain stability, especially in the case of dried products, it is necessary to choose the optimal barrier properties of the material depending on the sensitivity of the packed product (Gvozdenović 1981; Gvozdenović & Curaković, 1989, 1993, 1995; Heiss, 1980; Pöökkönen & Matilda, 1991).

Powdered products are very sensitive during storage. The change of characteristic colour is very quick, resulting in loss of colour or darkening. The active surface and water content of the product are detrimental factors which emphasize the change of colour intensity (Köul, 1980).

Water activity of powdered products is low (Heiss, 1989; Heiss & Eichner, 1971), and these products are in the range of maximal affect of nonenzymatic darkening. According to a number of authors, water activity affects the non-enzymatic changes and darkening reactions of products. (Eichner & Ciner-Doruk, 1979; Eichner & Karel, 1972; Karel & Labuza, 1968; Karel, Proctor & Wiseman, 1979; Labuza, 1973, 1975; Labuza & Saltmarch, 1980; Loncin, Bimbenet & Lenges, 1968). Their conclusions are supported by investigations of powdered raspberry, tomato, carrot, celery and onion colour change (Gvozdenović, 1981; Gvozdenović et al. 1988, 1993, 1995, 1997).

Daravingas and Cain (1966), report the degradative effect of sugar due to interaction of colour pigments from the product and sugar or active sugar degradation products. Bošković (1972) and Schoeber, Tannenbaum and Labuza (1969), report that reducing sugars, produced at low water activity, are potential reactants in the darkening process. Gvozdenović et al. (1987, 1993, 1995, 1997), Eichner (1975), Labuza (1973) and Netto (1997) conclude that initial quality and storage conditions, as well as moisture content, partial oxygen pressure in the packaging, permeability of packaging materials and storage time, significantly affect the colour change.

# 2. Materials and methods

An industrially produced powdered product selected by natural aroma and the colour of orange, was used for the investigations. Fifty per cent of sugar and moisture stabilizer was added to the product. Different combinations of packaging materials used in the industry for the packing of powdered products were used.

- 1. Combination PAP/PE, (paper/polyethylene) made of paper, surface mass 50  $g/m^2$ , laminated with 20  $\mu$ m polyethylene layer.
- 2. Combination PAP/ALU/PE (paper/aluminiumpolyethylene), made of 110  $g/m^2$  paper, 7  $\mu$ m aluminium foil, and 20  $\mu$ m polyethylene, produced by a lamination process.
- 3. Combination  $PET_{met}/PE$  (metalyzed polyester/ polyethylene), made of 12  $\mu$ m metalized polyester, laminated with 40 um polyethylene.
- 4. Combination PET/ALU/PE (polyester/aluminium/ polyethylene), made of 12 um polyester, 7 um aluminium foil and 50 µm polyethylene, produced by lamination.

Light permeability of combined materials was determined on a UV spectrophotometer UNICAM-SP 800 (JUS G.C8.511, Gvozdenović, 1981, 1987), while the permeabilities of air and water vapour were determined by applying the method according to Lyssy, DIN 53380 and DIN 53122, on GPM-200 and Vapor Permeation Tester L-80.

Colour characteristics of powdered orange base juice were determined instrumentally on the tristimulus photocolorimeter MOM-color 100. The results are presented in the CIE-system. The average reflectance or brightness e.g. darkening of colour as  $y(\%)$  was measured and the change of dominant wavelength (nm) was followed.

In the CIE-system, the dominant wavelength (nm) of the investigated sample is determined on the bases of values:  $x_1$ ,  $x_2$ , y and z obtained on the tristimulus photocolorimeter MOM-color 100. The tristimulus coefficients  $X$  and  $Y$  can be calculated using the formulae

$$
X = x/(x + y + z) \text{ and } Y = y/(x + y + z)
$$

where  $x = x_1 + x_2$ 

The  $X$  and  $Y$  values in the chromaticity diagram according to CIE (1986) determine the point F, which is connected with point C, obtained by adjusting the photometer with the standard. The CF line is prolonged and the intersection with the contours of the diagram (Diag. 1) represents the dominant wavelength.

After the investigation of initial colour quality, the packaging units were formed on the packing machine  $MPK-1$ , El FMO Niš. The packing and closing of samples were performed under normal atmospheric pressure (samples 1A, 2A, 3A and 4A) and under vacuum on the Audio Electron machine (samples 1V, 2V, 3V and 4V).



Diagram 1. Determination of dominant wavelength according to CIE (1986) (chromaticity diagram).

The change of characteristic colour was determined after 10 days of storage, and then after 1, 3, 6, 9 and 12 months of storage at room temperature  $(20 \pm 5^{\circ}C)$ .

## 3. Results and discussion

The permeabilities of light, air and water vapour of chosen combinations were investigated in order to determine the barrier properties of packaging materials. The permeability of air and water vapour is presented in Table 1.

The combinations with paper and aluminium are practically impermeable to light, while the combination  $PET<sub>met</sub>/PE$  is characterized by partial light permeability in the UV range of the spectrum. The combinations with aluminium (PAP/ALU/PE and PET/ALU/PE) are practically impermeable to water vapour and air, while the barrier characteristics of  $PET_{met}/PE$  are better, e.g. lower permeability values to air and water vapour molecules than the PAP/PE combination. This is quite understandable, bearing in mind the porous structure





of paper as the packaging material and thin layer of polyethylene.

# 3.1. Investigation of average reflectance (brightness or colour darkening)

The change of characteristic average reflectance is presented in Fig. 1.

The average reflectance of all analyzed samples after the packing was the same,  $y=24.66\%$ . After packing, and storage for 10 days, a significant decrease of average reflectance was recorded: 19.18, 18.47, 19.12 and 17.88% for samples IA, 2A, 3A and 4A, respectively. In comparison to the 0 day (packing day), during storage from the 30th till the 90th day, a significant stabilization of average reflectance was noticed. The average reflectance of samples further decreased and, after 270 days it was: 15.19, 16.26, 16.88 and 16.12% for samples 1A, 2A, 3A and 4A, respectively. The values significantly decreased to 12.00, 12.01, 12.20 and 12% (for the same samples) after 360 days of storage, compared to the packing day, when the colour of the instant product was optimally "light (bright)", now the colours were expressively "dark". After 10 days of storage of samples packed under vacuum, PAP/PE (lV), PAP/ALU/PE (2V),  $PET_{met}/PE$  (3V) and  $PET/ALU/PE$  (4V), the average reflectance decreased slightly and the measured values (y %) were: 21.29, 21.54, 21.09 and 20.74%, respectively. After 30 days of storage the measured values for the same samples were: 17.68, 17.81, 18.77 and  $16.63\%$ . The decrease trend of average reflectance in vacuum packed samples was smaller than in samples packed under atmospheric pressure (IA, 2A, 3A and 4A).



Fig. 1. Average reflectance.  $1A - PAP/PE -$  under atmospheric pressure;  $2A - PAP/ALU/PE -$  atmospheric pressure;  $3A - PET_{met}$  $/PE$   $-$  atmospheric pressure;  $4A$   $-$  PET/ALU/PE  $-$  atmospheric pressure;  $1V - PAP/PE - vacuum$ ;  $2V - PAP/ALU/PE - vacuum$ ;  $3V - PETmet/PE - vacuum$ ;  $4V - PET/ALU/PE - vacuum$ .

A more significant decrease of average reflectance was noticed in samples 1V, after 180 days of storage, and the measured value was  $y=12.78\%$ . No changes of average reflectance were noticed in samples 2V, 3V and 4V. Similar results were obtained after 360 days of storage, when the biggest decrease of values was again noticed in sample 1V and the average reflectance was  $y=11.02\%$ . The values after 360 days of storage for samples 2V, 3V and 4V were insignificantly different compared with the values obtained after 180 days, and were the following:  $2V - 17.20\%$ ,  $3V - 16.34\%$  and  $4V - 17.00\%$ .

# 3.2. Mathematical model of average reflectance

In order to describe average reflectance change during time, as welt as to predict system behaviour, the mathematical model was defined for all examined cases. Therefore, a type of correlation was chosen and its parameters were determined by the least squares approach. The exponential decay type of correlation was chosen. More complex cases (samples  $1A-4A$  and 1V) were solved by using the following equation:

$$
y = y_0 + A_1 \exp(-(x - x_0)/t_1) + A_2 \exp(-(x - x_0)/t_2)
$$
  
+  $A_3 \exp(-(x - x_0)/t_3)$ 

where  $y_0$   $x_0$ ,  $A_1$   $A_3$  and  $t_1$ -t<sub>3</sub> are the parameters that will be defined by the processing of experimental data. Eight unknown parameters require at least eight measurements. As the total number of measurements was seven, in all examined cases, at least one point was added before the processing started. It was done so that the trends of changes remained as the measured ones.

The dependent variable  $(y)$  is the average reflectance while the independent variable  $(x)$  is the time period.

Slightly simple cases (samples 2V - 4V) were modelled by applying the simplified exponential equation as follows:

$$
y = y_0 + A_1 \exp(-(x - x_0)/t_1) + A_2 \exp(-(x - x_0)/t_2)
$$

Graphical presentation of the correlations can be seen in Figs. 2-9, while their statistical characterization is given in Tables 2–9.

Description of the average reflectance change by the exponential correlation, in terms of time period, was very successful. Such a description shows very fast decrease of y-values at the beginning of the experiment. This is typical of all examined samples as is obvious from the fitting curves (Figs. 2–9). The analysis of  $t_1$ values (which are rather low) proved the conclusion. After the period of fast decrease, of period of slower decrease takes place for all samples except those exposed to vacuum ( $2V-4V$ ). The average reflectance



Fig. 2. Experimental and calculated averages reflectance (1A- sample).



Fig. 3. Experimental and calculated average reflectance (2A- sample).



Fig. 4. Experimental and calculated average reflectance (3A- sample).



Fig. 5. Experimental and calculated average reflectance (4A- sample).



Fig. 6. Experimental and calculated average reflectance (1V- sample).



Fig. 7. Experimental and calculated average reflectance (2V- sample).



Fig. 8. Experimental and calculated average reflectance (3V- sample).



Fig. 9. Experimental and calculated average reflectance (4V- sample).

## Table 2 Mathematical model of average reflectance in terms of time period (1A-sample)

	Parameter	Error
$y_0$	$-217.01118$	53.41305
$x_0$	$\theta$	****
A <sub>1</sub>	5.77091	0.56742
t <sub>1</sub>	3	****
A <sub>2</sub>	$-17.55809$	6.53522
$t_2$	360	****
$A_3$	253.46217	59.68273
$t_3$	5000	****

Table 3

Mathematical model of average reflectance in terms of time period (2A-sample)

Mathematical model: expdecay $Chi2 = 072156$		
	Parameter	Error
$y_0$	$-301.3994$	108.47079
$x_0$	0	****
A <sub>1</sub>	7.14591	1.15232
t <sub>1</sub>	3	****
A <sub>2</sub>	$-30.93678$	13.27168
t <sub>2</sub>	360	****
$A_3$	349.8693	121.203
$t_3$	5000	****

for vacuum samples remains constant until the end of the experiments. However, in the case of air samples and the sample IV, decrease of the average reflectance became faster at the end of the experiments, so that the last measured value is approximately 12%. Measured minimum of vacuum samples is much higher, i.e., it is near to 17%.

# 3.3. Change of dominant wavelength

The change of dominant wavelength of powdered orange base juice is presented in Table 10 and Figs. 10 and 11.

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Mathematical model of average reflectance in terms of time period (3A-sample)



Table 5 Mathematical model of average reflectance in terms of time period (4A-sample)

Mathematical model: expdecay $Chi2 = 0.50297$		
	Parameter	Error
$y_0$	$-413.91$	90.15
$x_0$	0	****
$A_1$	7.61	0.95
t <sub>1</sub>	3	****
A <sub>2</sub>	$-43.38$	11.14
$t_2$	360	****
$A_3$	474.36	100.8
$t_3$	5000	****

Table 6

Mathematical model of average reflectance in terms of time period (1V-sample)

Mathematical model: expdecay $Chi2 = 0.71517$		
	Parameter	Error
$y_0$	$-359.9$	128.6
$x_0$	$\Omega$	****
A <sub>1</sub>	7.57	1.67
t <sub>1</sub>	15	****
A <sub>2</sub>	$-34.92$	16.63
t <sub>2</sub>	360	****
$A_3$	411.9	144.14
$t_3$	5000	****

Table 7

Mathematical model of average reflectance in terms of time period (2V-sample)

The colour of samples 1A and 3A was red-orange (RO) (615 nm) until the 30th day of storage. Further, up to 270 days of storage, the colour of these samples shaded into orange (O); e.g. the dominant wavelength was 588 nm. After 270 days, the colour of sample 1A shaded into the yellow-orange part of the spectrum (582 nm) and, at the end of the storage (360th day), the dominant wavelength increased to 615 nm; the colour of these samples shaded into a red-orange nuance. In sample 3A, the dominant wavelength, after 270 days of storage, was 615 nm, a red-orange nuance, and it remained unchanged until the end of storage.

The colours of samples 2A and 4A were in the orange range of the spectrum from the 10th to the 270th day of

Table 8 Mathematical model of average reflection (3V-sample)

Mathematical model: $y = y_0 + A_1 \hat{e}(-(x - x_0/t_1) + A_2 \hat{e}(-(x - x_0)/t_2))$	
Chisgr = $0.75147$	
X offset $(x_0) = 0$	
Y offset $(v_0) = 15.8168$	
$t_1 = 8.264 A_1 = 4.804$	
$t_2 = 139.5 A_2 = 4.049$	

Table 9

Mathematical model of average reflectance in terms of time period (4V-sample)

Mathematical model: $y = y_0 + A_1 \hat{e}(-(x - x_0/t_1) + A_2 \hat{e}(-(x - x_0)/t_2))$
Chisqr = $0.14094$
X offset $(x_0) = 0$
Y offset $(v_0) = 17.1175$
$t_1 = 16.92 A_1 = 11.17$
$t_2 = 41.34 \text{ A}_2 = -3.545$

storage. After 30 days, the dominant wavelength was 592 nm (orange nuance) and further, up to the 180th day, it slightly decreased (588 nm), but still remained in the orange range of the spectrum. At the end of the storage period, the colour of all samples shaded into a red-orange nuance.

The dominant wavelength of samples packed under vacuum, during the whole storage period, was in the range 586-592 nm, and the colour was in the orange part of the spectrum, with the exception of sample 1V. After 270 days, the colour of sample 1V shaded into a yellow-orange nuance (582 nm). The results obtained, during the investigation of dominant wavelength of instant orange-based product, confirmed the advantages of packing under vacuum. The colour characteristics of samples packed in combined materials PAP/ALU/PE, PET<sub>met</sub>/PE and PET/ALU/PE remained unchanged during the whole storage period.

In samples packed in PAP/PE, due to increased permeability of this material to air, vacuum decreased and the partial pressure of air increased in the packaging units. This caused the fading of samples and appearance of a yellow-orange nuance and significantly greater changes of colour quality compared to the samples packed in the same combination of packaging materials but under atmospheric conditions. This is confirmed by the values of average reflectance, i.e. the colour darkening during longer storage of samples.

The oscillation cyclicity of colour nuances can be explained by changes of spectral colour characteristics, due to formation of non-enzymatic degradation products. The intensity of change depends on the applied combinations of materials and packing conditions, (Eichner & Karel, 1972; Heiss, 1980; Loncin et al.,







#### Time (days)

Fig. 10. Dominant wavelength.  $1A - PAP/PE$  — under atmospheric pressure;  $2A - PAP/ALU/PE -$  atmospheric pressure;  $3A - PET$  $met/PE$   $-$  atmospheric;  $4A$   $PET/ALU/PE$   $-$  atmospheric pressure:  $1V - PAP/PE - vacuum$ ;  $2V - PAP/ALU/PE - vacuum$ ;  $3V - PETmet/PE - vacuum$ ;  $4V - PET/ALU/PE - vacuum$ .



Fig. 11. Dominant wavelength according to the CIE system.

1968). The increase of moisture content in dried products results in decreased nonenzymatic darkening due to dilution of reactants or formation of intermediates insoluble in water. The investigations of dehydrated raspberry colour changes support previous work (Gvozdenović, 1987; Guozenović & Curaković, 1993, 1995).

## 4. Conclusion

The changes of an orange based powdered product show that, besides the packing conditions, which eliminate the influence of incorporated oxygen, the quality of the packaging materials and their combinations significantly influence the colour change. The large active surface of the powder intensifies the non-enzymatic changes. The powder is in the optimal range of water activity, for the nonenzymatic processes.

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