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DIETARY USE OF RED BEET CRUNCHABLE TABLETS OF FREEZE-DRIED RED BEET PULP

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ABSTRACT

The formulation of crunchable tablets of freeze-dried red beet pulp, a material of therapeutic and dietary value, is described. Various diluants, binders, lubricants, sweeteners and flavoring were chosen to provide desirable organoleptic properties, adequate stability and convenient manufacturing characteristics. 13 different formulations were tested and appraised by a panel for acceptability.

INTRODUCTION

The beneficial effects on man of dietary fiber have attracted considerable attention in recent years. Fiber is implicated in the regulation of gastro-intestinal transit, in particular through retention of fluid, and is useful in the treatment of a number of conditions : digestive (constipation, diverticulosis, functional colopathies), vascular (hemorrhoids, sigmoid varicose

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veins) and metabolic (obesity, diabetes, blood lipid imbalance, atherosclerosis, cholesterol-induced gall stones) (1-4).

In the course of work on the dietary uses of red beet (5, 6) we became interested in red beet pulp. This material is rich in cellulose fiber and can strongly stimulate transit. Pharmacological studies in conscious rats showed moderate acceleration of transit after oral administration of pulp at 400 $mg.kg^{-1}$ per day (7). Extrapolating this figure to a man weighing 60 kg to 80 kg gives a daily dose of 2 g to 3 g of pulp. We investigated the possibilities of incorporating pulp in crunchable tablets, a convenient and highly acceptable form, along with appropriate flavoring to mask the taste of the beet. Red fruit flavoring (raspberry - blackcurrant) was chosen, coupled with an appropriate sweetener.

MATERIALS AND METHODS

Raw Materials

Preparation of the Active Ingredient. Freeze-dried Red Beet Pulp

Raw red beetroots (*Beta vulgaris*, Globe variety, Laura race) were washed and pulped with a knife-breaker (Stephan). The pulp was spread on plates in layers not more than 20 mm thick, deep-frozen and freeze-dried (48 h in a Sublivac CS 3P freeze-drier). The operating efficiency was about 16 %. The freeze-dried pulp was crushed (Turmix screw-breaker) and screened through a sieve of mesh size 500 µm (AFNOR Tamisor).

Numerical Color Identification of Freeze-dried Red Beet Pulp

This was performed by reflectance using a Minolta CR-200 Chroma meter (8) by tristimulus colorimetry : L represents value, while hue and chroma are expressed as a and b respectively, a corresponding to a red-green color axis and b to a yellow-blue color axis.

Rheological Properties of Freeze-dried Red Beet Pulp

These were quantified using a Hosokawa powder characteristics tester (9), designed mechanically to measure the compressibility, angle of repose, spatula angle, fall angle and dispersibility of a

powder. These parameters, along with particle size distribution, serve to determine the flowability index and the floodability index of the powder, which are a measure of its ease of handling.

Excipients for Compression and Additives

Diluants : sucrose, lactose, corn starch and mannitol (Coopération Pharmaceutique Française)

Binders : β-cyclodextrin (Roquette) and hydroxymethylcellulose, Metolose SH 15000® (FMC Corp. Seppic).

Lubricants : talc, magnesium stearate and hydrogenated vegetable oil, Lubritab®.

Sweeteners and flavorings : aspartam (Searle), anhydrous citric acid, ascorbic acid (Prolabo), liquid caramel for foods, artificial raspberry flavoring 6K103 and natural condensed blackcurrant flavor 11E250 (IFF).

Crunchable Tablets

Preparation

Tablets containing 20 % freeze-dried red beet pulp weighing 2.20 g were prepared according to the formulations set out in Tables 1 and 2. Preparation was by the wetting granulation method. The raw materials were screened through a 500 μm sieve (AFNOR Tamisor) and blended for 20 min in a cubical mixer (Erweka). The wetting solution was 60 % ethanol in which the flavoring had been dissolved. This was added to the powder mixture in a planetary mixer (Hobart) and blended until uniform. The resulting mixture was screened through a 1,6 mm sieve held in an oscillating granulator (Erweka). The wet granules were dried for 40 min in a fluidized bed (Aeromatic) at 50°C. The dried product was screened through a 1 mm sieve (Erweka), blended with the lubricant for 10 min in the cubical mixer (Erweka) and finally tabletted in a Korsch tabletting machine fitted with 20 mm flat-faced punches.

Properties of Finished Tablets

Mechanical Properties. Crushing strength was measured with a Stokes apparatus. Each test was carried out on a sample of 10 tablets taken at random from the production batch. Friability was measured

TABLE 1

Tablet Formulations Containing β-Cyclodextrin as Binder

Nr. Formulations	1	2	3	4	5
Excipients (%)					
Freeze-dried Red beet	20	20	20	20	20
Sucrose	30	30	30	30	
Lactose	15	15	15	15	39
Mannitol	16.5	16.6	16	16.1	22
Corn starch	17	11	10.7	11	11
β-Cyclodextrin	2.5	2.5	2.5	2.5	2.5
Raspberry Flavour	0.5	0.5	0.5	0.5	0.5
Blackcurrant Flavour	1.5	1.5	1.5	1.5	1.5
Caramel	0.8		0.8		
Citric acid	1	1	1	1	1
Ascorbic acid	0.5	0.5	0.5	0.5	0.5
Aspartam		0.4		0.4	0.4
Talc	0.5	0.5	0.5	0.5	0.5
Magnesium stearate	0.5	0.5			
Lubritab®			1	1	1

TABLE 2

Tablet Formulations Containing Metolose 90 SH 15000® as Binder

Nr. Formulations	6	7	8	9	10	11	12	13
Excipients (%)								
Freeze-dried Red Beet	20	20	20	20	20	20	20	20
Sucrose	30	30	30	30	30	30	30	
Lactose	15	15	15	15	15	15	15	39
Mannitol	16.5	16.6	16.3	16.5	16	16.1	16.5	22
Corn starch	10.7	11	11	11	10.7	10.7	11	11
Metolose 90 SH 15000®	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Raspberry Flavour	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Blackcurrant Flavour	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Caramel	0.8		0.3		0.8	0.3		
Citric acid	1	1	1	1	1	1	1	1
Ascorbic acid	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Aspartam		0.4	0.4	0.5		0.4	0.5	0.5
Talc	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Magnesium stearate	0.5	0.5	0.5	0.5				
Lubritab®					1	1	1	1

with a Roche friabilator. This apparatus subjects a sample of 10 tablets to friction and impacting for 5 min. The weight loss of the sample is measured and ideally should not exceed 2 %. Weight uniformity was assessed on a sample of 20 tablets using a precision balance (10).

Macroscopic and Organoleptic Properties

Macroscopic Properties. The uniformity of the tablets was checked by visual inspection. Their appearance and surface coloration was quantified with the Minolta CR-200 Chroma meter.

Organoleptic Properties. A panel of ten adults was asked to appraise the degree of masking of the red beet taste, and the flavoring. Suitable flavoring was considered as that which is neither repellent nor so attractive that it may induce compulsive overconsumption. The olfactory qualities of the sample, its crunchability and taste parameters on a six-point scale (very bad, bad, rather bad, fairly good, good, very good (11), were evaluated by the panel for acceptability. The panel were asked to make suggestions for improvement of organoleptic properties.

Stability of the Dosage Form

The stability at 22°C of the freeze-dried red beet pulp in tablets both exposed to and protected from light was evaluated by spectrophotometry. The assay method was as follows :

A tablet was dissolved in 150 ml of distilled water in an ultrasonic tank (Bransonic), the solution was spun for 15 min at 5000 rpm and the supernatant filtered on bleached filter paper (Prolabo). The spectrophotometric absorption of the colored solution was measured at 538 nm, the maximum absorption wavelength of betanin. The results are expressed as percentage of intact pulp against time.

RESULTS AND DISCUSSION

Properties of Freeze-dried Red Beet Pulp

Numerical Color Identification

The three values for the perceived color parameters were

L = 32.29 ; a = + 26.11 ; b = + 1.31

Rheological Properties

Flowability Parameters

The values obtained are set out in Table 3.

TABLE 3

Rheological Properties of Frieeze-dried Red Beet Pulp
Flowability Parameters

Apparent density (g/cm^3)			Compressibility		Angle of repose		Spatula angle		Uniformity		"Flowability"
Aerated (A)	Packed (P)	Mean ($\frac{A+P}{2}$)	(%)	Index	Degree	Index	Degree	Index		Index	Total Index
0.41	0.58	0.495	29	12	42	16	59	16	9	19	63

Compressibility. Compressibility was 29 %. The value beyond which flowability is adversely affected is 40 %. The value above which a tendency to flushing will occur is 20 %.

Angle of Repose and Spatula Angle. The angle of repose was 42°, and the spatula angle was 59°. Flowability is considered poor when the angle of repose exceeds 45° and/or when it is less than the spatula angle. Hence, the flowability of the freeze-dried red beet pulp appeared less than ideal.

Particle Size Distribution : Uniformity. A measure of the uniformity of particle size was obtained by determining the particle size distribution by the vibrating sieve method (intensity 5, 86 seconds). 5 g of powder was shaken successively on five sieves 75 mm in diameter of mesh size 350, 250, 150, 75 and 45 µm. The proportion of material passing through was plotted against the sieve mesh size. From these plots, the uniformity index was calculated as the ratio of mesh size at 60 % to that at 10 % fall-through (figure 1). This index can vary from 1 to 35 (Hosokawa standards). The narrower the particle size range, the closer the uniformity index is to 1 and the freer flowing the powder is. The value found here for freeze-dried red beet pulp was 9, which was indicative of a "good" uniformity.

The total index of flowability of freeze-dried red beet pulp was 63. The range of normal flowability is 60 to 69.

Floodability Parameters

The values obtained are set out in Table 4.

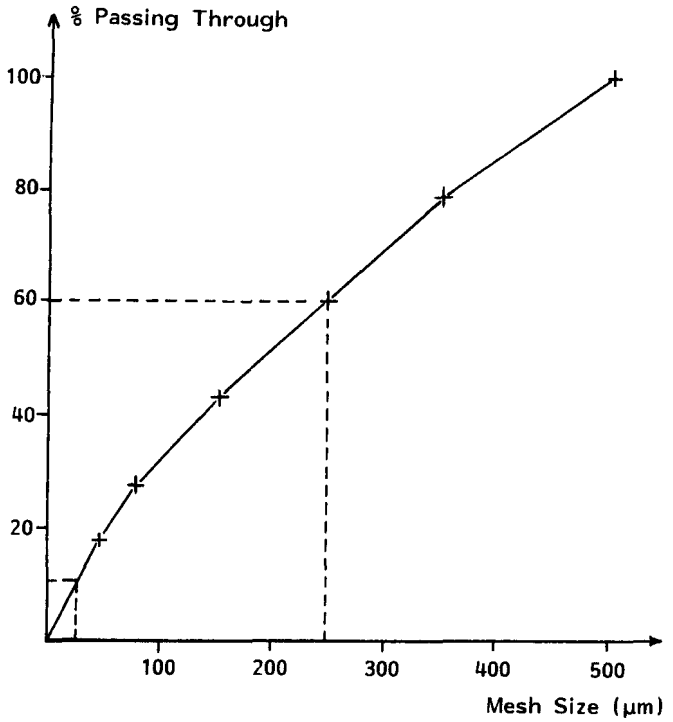

FIGURE 1

Particle Size Distribution of Freeze-dried Red Beet Pulp

TABLE 4

Rheological Properties of Freeze-dried Red Beet Pulp
Floodability Parameters

"Flowability"		Fall angle (°)		Difference angle (°)		Dispersibility	"Floodability"	
Index Table 3	Index	Degree	Index	Degree	Index	(%)	Index	Total Index
63	25	18	24	24	20	10	9.5	78.5

Fall Angle and Difference Angle. According to the Hosokawa standards, the implications of fall angle for the fluidity of a powder are optimally favorable at 10° and adverse beyond 64°. The optimal value for the difference angle is 30°. For freeze-dried red beet pulp, we found a fall angle of 18° and a difference angle of 24°. These values thus indicate favorable floodability.
Dispersibility. A dispersibility above 50 % indicates that the powder has a strong tendency to flush. Here, freeze-dried red beet pulp gave a value of 10 %.

Total index of floodability of freeze-dried red beet pulp was 78.5. This is in the range (60 - 79) considered as fairly high.

Properties of Finished Tablets

Mechanical Properties

Mean crushing strength : 9 kg : favorable
Friability : below 1 % : favorable.
Weight uniformity : 2.20 g ± 5 % : in conformity with French Pharmacopoeia standards.

Macroscopic and Organoleptic Properties

Numerical Color Identification

Quantification of the surface color of the tablets gave similar L, a and b values for all the formulations. L lay between 60 and 63, a lay between + 19 and + 20 for formulations 1, 3, 5, 6, 7 and 8 containing caramel, and between + 15.5 and + 16.5 for the others. b lay between - 0.3 and + 0.5. The decrease in the values of a and b is due to the presence of white excipients. The high value of L indicates a shiny surface.

Organoleptic Properties

Tablets Containing β-Cyclodextrin as Binder. The first two formulations both contained the classical combination of talc and magnesium stearate as lubricant but differed in the sweetener used.

Formulation Nr 1 : This formulation was technically convenient and acceptable organoleptically.

Formulation Nr 2 : In this formulation, caramel was replaced by aspartam as sweetener, essentially for dietary reasons. Its acceptability was as for formulation Nr 1.

Formulations Nr 3 and Nr 4 : In these, magnesium stearate was replaced by 1 % Lubritab®. This change of lubricant slightly improved compressibility, and did not affect the organoleptic acceptability of the tablets.

Formulation Nr 5 : A low-calorie preparation suitable for diabetics was obtained by eliminating sucrose. The sweetening power of not only aspartam (180 - 200) but also of the excipients lactose (0.25) and mannitol (0.5) were made use of. The technical, macroscopic and organoleptic qualities of this formulation were particularly favorable.

Tablets Containing Metolose 90 SH 15000® as Binder

Formulation Nr 6 : Compared with formulation Nr 1, the change of binder did not alter the organoleptic properties of the tablets.

Formulation Nr 7 : The panel found these tablets slightly lacking in sweetening compared with formulation Nr 2. This may be because β-cyclodextrin tastes sweeter than Metolose®.

Formulations Nr 8 and Nr 9 : Wichever the sweetener, caramel 0.3 % - aspartam 0.4 % or aspartam alone 0.5 %, the tablets were highly acceptable organoleptically.

Formulations Nr 10, Nr 11 and Nr 12 : As above, Lubritab® in place of magnesium stearate improved compressibility without adversely affecting the organoleptic qualities.

Formulation Nr 13 : This sucrose-free formulation was technically acceptable, and had macroscopic and organoleptic properties that were slightly more favorable than formulations Nr 10, Nr 11 and Nr 12.

All the formulations, except Nr 7 that lacked sweetening, were thus acceptable technically and organoleptically.

Stability of Freeze-dried Red Beet Pulp in Tablets

Tables 5 and 6 shows the percentage of intact red beet pulp against time in tablets stored at ambient temperature respectively in and away from light.

TABLE 5

Stability of Freeze-dried Red Beet Pulp in Tablets Exposed to Light

Nr Formulations / Time (months)	1	2	3	4	5	6	8	9	10	11	12	13
0	100	100	100	100	100	100	100	100	100	100	100	100
3	88	88	87	87	88	85	85	86	86	84	84	85
6	81	80	75	76	77	73	74	72	71	70	69	72
9	76	78	71	70	72	68	69	67	65	66	64	67
12	70	71	65	66	68	65	65	64	60	61	60	62

TABLE 6

Stability of Freeze-dried Red Beet Pulp in Tablets Stored in the Dark

Nr Formulations / Time (months)	1	2	3	4	5	6	8	9	10	11	12	13
0	100	100	100	100	100	100	100	100	100	100	100	100
6	100	100	100	100	100	99	100	100	98	97	98	98
9	100	100	92	93	95	91	95	96	90	90	91	93
12	97	98	90	88	90	89	93	92	81	80	82	90

Formulations Containing β-Cyclodextrin

Formulations Nr 1 and Nr 2 were more stable when exposed to light than Nr 3. Nr 4 and Nr 5 (70 % and 71 % against 65 %, 66 % and 68 % intact active material after one year). Hence magnesium stearate is to be preferred to Lubritab® as lubricant.

Formulations Containing Metolose®

Formulations Nr 6 to Nr 13 that use Metolose® as binder were as stable or slightly less stable than formulations Nr 1 to Nr 5 containing β-cyclodextrin. Here as above, tablets containing magnesium stearate were more stable than those made up with Lubritab®. In light-exposed tablets, formulations Nr 6, Nr 8 and Nr 9 had retained 64 % to 65 % of their active material after one year, against 60 % to 62 % for formulations Nr 10 to Nr 13. In tablets kept in the dark, the pattern was similar, with formulations Nr 1 and Nr 2 being the most stable, followed by formulations Nr 6, Nr 8 and Nr 9.

CONCLUSIONS

The results of macroscopic and organoleptic appraisal and stability measurements show two formulations to be of particular interest, both containing β-cyclodextrin and lubricated with talc and magnesium stearate, but differing in the sweetener used, caramel in formulation Nr 1 and aspartam in Nr 2. Formulation Nr 5, though slightly less stable, has the advantage of being sucrose-free. Despite their slightly lower stability, formulations Nr 6, Nr 8 and Nr 9 containing Metolose 90 SH 15000®, talc and magnesium stearate still perform well.

Thus crunchable tablets of freeze-dried red beet pulp having highly acceptable macroscopic and organoleptic properties can be produced without technical difficulty, providing a useful form for transit regulation.

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REFERENCES

1. F. Lamisse, C. Couet, Y. Bacq, T. Constans and J. Delarue, Cah. Nutr. Diét., 22, 5, 397 (1987).
2. C. Laval, "Les fibres alimentaires et leur utilité en thérapeutique humaine", Thesis Doct. Pharm., Paris XI, 1986.
3. Z. Madar and R. Thorne, in "Progress in Food and Nutrition Science", Pergamon Journals Ltd., 1987, p. 153.
4. J. L. Slavin, J. Am. Diet. Assoc., 87, 9, 1164 (1987).
5. A. Arvouet-Grand, B. Lejeune, A. Pourrat and P. Bastide, S.T.P.-Pharma, 5, 6-7, 448 (1989).
6. A. Arvouet-Grand, B. Lejeune and A. Pourrat, Pharm. Acta Helv., accepted Nr. 875 (Sept. 1989).
7. A. Arvouet-Grand, "Etude pharmacologique et pharmacotechnique de la betterave rouge", Mémoire Doct. Rech. Univ., Clermont-Ferrand, 1989.
8. Minolta, "Precise Color Communication", Technical Documentation, 1988.
9. Hosokawa, "Powder Characteristics Tester", Operating Instructions.
10. Pharmacopée Française, Xe édition, Maisonneuve, Paris, 1983.
11. E. Gajda, "Aromatisation des médicaments. Application à un sirop antitussif", Thesis D. Pharm., Clermont-Ferrand, 1986.