



STUDIES ON LOW CALORIE ICE MILK
4-PREPARATION OF LOW CALORIE
FROZEN YOGHURT

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Abstract

This study was carried out to manufacture a low calorie frozen yoghurt. The base formula contained 2% fat, 13% milk solids not fat and 0.7% gelatine. Treatment 1 contained 15% sucrose (control) while treatment 2 contained 13% sucrose and 2% sorbitol. To each treatment 2% starter and suitable amount of vanilla were added. The mix of low calorie frozen yoghurt was analysed for acidity, specific gravity, weight per gallon and freezing point. The resultant low calorie frozen yoghurt was analysed for specific gravity, weight per gallon, overrun and melting resistance. Also organoleptic evaluation was done. Acidity and overrun were increased in the presence of sorbitol. Specific gravity of the mix as well as the resultant low calorie yoghurt was decreased in the presence of sorbitol. Also freezing point decreased as sorbitol was added. The difference between treatments in the organoleptic properties was not significant. This study concluded that, an acceptable and palatable frozen yoghurt can be made by using sorbitol as a partial substitute of sucrose.

Introduction

Kosikowski (1) defined frozen yohurt as a flavoured yoghurt which is frozen in ice cream freezers with the introduction of air to give about 50% overrun. Knupp(2) reported that frozen yoghurt is a dairy light meal desert that has all of the refreshing qualities of sherbet, combined with an unusual tanginess and concentrated

flavour character contributed by the presence of the cultured yoghurt.

Kosikowski (1) reported two very low calorie formulas for the preparation of frozen yoghurt. One of them is for making a raspberry- flavoured yoghurt more closely to the nutritional properties of standard yoghurt. The second formula is for making a plain frozen yoghurt. He also explained the process of a suitable plain frozen yoghurt of about 75 cal/100g with less than 1% lactose.

Bray (3) stated that, frozen yoghurt could be produced from mixes containing hydrolysed whey, in which the lactalbumin acts as a stabilizer and emulsifier. The enzymic splitting of lactose into glucose and galactose enhances the sweetness and reduces the need for sucrose. Also he mentioned that there are different ways of adding fruit to frozen yoghurt.

Mcgregor and White (4) studied the effect of sweeteners on the quality and acceptability of yoghurt. They manufactured sweetened low fat plain and swiss-style fruit flavoured yoghurts using cream and reconstituted non fat dry milk. They concluded that as amount of sweetener increased, the time required to get pH 4.4 is increased.

This study was carried out to prepare a low calorie frozen yoghurt using sorbitol as a partial substitute for sugar.

Material and Methods

I- Materials:

Fresh cow's milk used in this study was obtained from the herd of faculty of agriculture, Minufia University, Egypt. Skim milk powder was an U.S.A. low heat product. Gelatine used was a pure grade produced by Adwic Labortary Chemicals, Cairo, Egypt. Sucrose and Vanilla were Egyptian products. Sorbitol used in this investigation was brand called, sorbitol Neosorb Requite P 11 420. Stabilizer used in this study was Palsgaard 5936 (emulsifier and stabilizer), produced by palsgaard Industrie AIS, Denmark.

II- Preparation of low calorie frozen yoghurt:

Low calorie frozen yoghurt was prepared using milk standardized to 2% fat, 13% solid non-fat, 0.7% gelatine, suitable amount of vanilla, starter [Loctobacillus bulgaricus and Streptococcus thermophilus] and two different kinds and concentrations of sugar as follow:

- Treatment no. 1 (control), contained 15% sucrose. Each batch from the mix weighted approximately (1kg)

was consisted of: standardized milk 817 gm, skim milk powder 32.68 gm, sucrose 127.45 gm, gelatine 6.84 gm and starter 16.34 gm.

- Treatment no. 2 contained 13% sucrose + 2% sorbitol. Each batch from the mix weighted approximately (1kg) was consisted of: standardized milk 817 gm, skim milk powder 32.68, sucrose 110.46 gm + sorbitol 16.99gm, gelatine 6.84 gm and starter 16.34 gm.

In each treatment, half amount of gelatine was added to the standardized milk then mixed very well, pasteurized at 72°C for 30 minutes and cooled to 30°C. The starter (Lactobacillus bulgaricus, Streptococcus thermophilus) was added to the above milk and mixed very well. The mix was placed in an electric controlled cabinet at (40-14°C) for 3 hours then cooled in a refrigerator at 5°C for 24 hours.

The other amount of gelatine was added to these ingredients (skim milk powder; sugars (sucrose, or a mixture of sucrose and sorbitol) and suitable amount of water) and mixed very well. The mix was pasteurized at 72°C for 30 minutes, cooled to 5°C and aged in the refrigerator at 5°C for 24 hours. Then the first part of (fermented milk) was added to the second part, mixed very well and suitable amount of vanilla was added.

Finally the mix was frozen in an experimental ice cream freezing machine [KAPRICE-CATTABRIGA]. This freezer was automatically controlled to stop whipping when ice cream reaches the right consistency. It was packaged in cups 100 gm, and placed in a cabinet held at - 25°C for hardening for 24 hours.

III- Methods of analysis:

- a- Mix properties: The mix was examined for acidity (5) specific gravity (6), weight per gallon (7,8) and freezing point according to the method recommended for milk by the FAO Regional Dairy Development and Training Centre for the Near East (9).
- b- Low calorie frozen yoghurt properties: Specific gravity (6), weight per (7,8), overrun (10), melting resistance (11) and organoleptic evaluation (12), were determined in the resultant low calorie frozen yoghurt.

The organoleptic evaluation was carried out by a regular panel from the Food Science Department, Faculty of Agriculture, Minufiya University. Evaluation was carried out according to Rothwell (12), for flavor (50 points), body and texture (40 points) and (10 points) for melting quality.

- c- Statistical analysis: The data were statistically analysed according to Steel and Torrie (13)."

Results and Discussion

a- Mix properties:

Effect of paratial replacement of sucrose with sorbitol on acidity, specific gravity, weight per gallon and freezing point of low calorie frozen yoghurt mixes are illustrated in table (1).

Acidity percentage in treatment 1 was 0.62%, while it was 0.65% in treatment 2.

The mean values of specific gravity (table 1) for treatments 1 and 2 were 1.1060 and 1.1030

The mean values of weight per gallon were 4.19 and 4.18 kg for treatments 1 and 2.

These results indicate that weight per gallon of mixes were closely related to the specific gravity of the corresponding mixes.

Freezing points were - 2.23°C and - 2.14°C for treatments 1 and 2 respectively (table 1). The freezing point increased very slightly in treatment 2 than treatment 1.

Table (2) shows that the T-test for the differences between treatments 1 and 2 in acidity, specific gravity,

Table (1) : Effect of partial replacement of sucrose with sorbitol on acidity, specific gravity, weight per gallon and freezing point of low caloric frozen yoghurt mixes.

Group comparison	Sucrose %	Sorbitol %	Starter %	Acidity %	Specific gravity	Weight per gallon (Kg)	Freezing °C
Treatment 1	15	--	2	0.62	1.1060	4.19	-2.23
Treatment 2	13	2	2	0.65	1.1030	4.18	-2.24

Table (2) : T-test results for comparison of treatments 1 and 2 in acidity, specific gravity, weight per gallon and freezing point of low caloric frozen yoghurt mixes.

Properties	t-value (Calculated)	t-value (Tabulated at 0.05 probability level)
(1) Acidity, comparison treatments 1 and 2.	1.19	2.78
(2) Specific gravity, comparison treatments 1 and 2	1.11	2.78
(3) Weight per gallon comparison treatments 1 and 2.	0.14	2.78
(4) Freezing point, comparison treatments 1 and 2.	1.42	2.78

weight per gallon and freezing point did not exceed the tabulated value at 0.05 level of probability.

b- Low calorie frozen yoghurt properties:

1- Specific gravity, weight per gallon and overrun:

The effect of partial replacement of sucrose with sorbitol on the specific gravity, weight per gallon and overrun of low calorie frozen yoghurt is shown in table (3).

The mean values of specific gravity for treatment 1 was 0.6420 and then slightly decreased to 0.6080 in treatment 2 due to the presence of 2% sorbitol (14). The mean values of weight per gallon were 2.43 and 2.30kg for treatments 1 and 2. From table (3) it could be seen that the overrun was 71.70% and 73.13% in treatments 1 and 2 respectively. The overrun was increased in treatment 2 and this increase is associated with the decrease of the specific gravity of low calorie frozen yoghurt. These results agree with that of Hofi et al. (15) and Omer (16). T-test for the difference between treatments 1 and 2 in specific gravity, weight per gallon and overrun did not exceed the tabulated value at the 0.05 level of probability table (4).

Table (3) : Effects of partial replacement of sucrose with sorbitol on specific gravity, weight per gallon and overrun of low caloric frozen yoghurt.

Group Comparison	Sucrose %	Sorbitol %	Starter %	Specific gravity	Weight per gallon (Kg)	Overrun %
Treatment (1)	15	--	2	0.6420	2.43	71.70
Treatment(2)	13	2	2	0.6080	2.30	73.13

Table (4) : T-test results for comparison of treatments 1 and 2 in specific gravity, weight per gallon and overrun of low caloric yoghurt.

Properties	t-value (Calculated)	t-value (Tabulated at 0.05 probability level)
(1) Specific gravity, comparison treatments 1 and 2.	0.94	2.78
(2) Weight per gallon, comparison treatments 1 and 2.	0.72	2.78
(3) Overrun, comparison treatments 1 and 2.	0.48	2.78

2- Melting resistance:

Table (5) shows the effects of partial replacement of sucrose with sorbitol on the melting resistance of low calorie yoghurt. The melting resistance is given as loss in weight percent of the initial weight of the tested sample at the end of 1, 1.5 and 2 hours. The average loss in weight at the first 60 minutes for treatments 1 and treatment 2 were 26.53% and 28.31% respectively. Both treatments showed a tendency to be highly resistance. It could be observed that treatment 1 has a high resistance than treatment 2. This may be due to the effect of sorbitol on the freezing point of low calorie frozen yoghurt mixes.

During the next 30 minutes the samples showed the same previous trend which occurred during the first period. Loss of weight was 42.62% and 46.81% for treatments 1 and 2 in the same order. At the last 30 minutes of the test the melted portions were 22.96% and 18.62% for treatments 1 and 2 respectively.

Table (6) shows that the T-test for the difference between treatments 1 and 2 melting resistance. After the first 60 minutes, and after the last 30 minutes it did not exceed the tabulated value at 0.05 level of probability; while after the second 30 minutes, it

Table (5) : Effect of partial replacement of sucrose with sorbitol on the melting resistance of low calorie frozen yoghurt.

Group Comparison	Sucrose %	Sorbitol %	Starch %	Loss per centage after the end of		
				First 60	Next 30	Last 30
Control (1)	12	-	2	26.53	42.62	22.96
(2)	13	2	2	28.31	46.81	18.62

Table (6) : T-test results for comparison of treatments 1 and 2 in melting resistance of low calorie frozen yoghurt.

Properties	T-value (Calculated)	t-value (Tabulated at 0.05 probability level)
Melting Resistance, comparison treatments 1 and 2 after :-		
(1) <u>First 60 minutes</u> :	0.08	2.78
(2) <u>Next 30 minutes</u> :	3.43	2.78
(3) <u>Last 30 minutes</u> :	1.57	2.78

exceeded the tabulated value at the same level of probability.

3- Organoleptic properties:

In this part samples 2 and 3 were the duplicate of the same sample and the taste panel did not know about it. The effect of sorbitol 2% with 13% sucrose on the organoleptic properties of low calorie frozen yoghurt is given in table (7). From these results, the score average for flavour were 40.7, 38.8 and 36.2 points for treatments 1, 2 and 3 respectively. Treatment 1 had a higher flavour score than treatments 2 or 3. The decrease in flavour score of treatments 2 and 3 may be due to the increase of acidity in the mixes, due to the presence of sorbitol which encourages the bacterial growth which produces acidity. The score averages of body and texture were 35.3, 34.2 and 34.5 points for treatments 1 to 3 respectively. Melting quality were 8.0, 7.5 and 7.8 for treatments 1, 2 and 3 respectively. These results indicated that the melting quality was decreased in treatments 2 and 3 than treatment 1. This decrease may be due the effect of sorbitol on the freezing point of low calorie frozen yoghurt. The total score was 84.0 for treatment 1 while was 78.8 and 78.5 for treatments 2 and 3 succeedingly.

Table (7) : Effect of sorbitol 2% with 13% sucrose on the organoleptic properties of low caloric frozen yoghurt.

Treatments No.	Sucrose %	Sorbitol %	Starter %	Flavour (50 points)	Body and texture (40 points)	Melting quality (10 points)	Total score (100 points)
Control (1)	15	--	2	40.7	35.3	8.0	84.0
(2)	13	2	2	38.8	34.2	7.5	78.8
(3)	13	2	2	36.2	34.5	7.8	78.5

Table (8) : Analysis of variance for organoleptic properties of low caloric frozen yoghurt.

Source of Variance	d.f	S.S	M.S	F	L.S.D
Blocks	5	812.4500	162.4900		
Treatments	2	114.1100	57.0550	2.85	-
Error	10	442.8900	44.2890		
Total	17				

The statistical analysis of variance (table 0) proved that the changes occurred in the total score of the resultant low calorie frozen yoghurt were not significant between treatments 1,2 and 3.

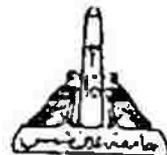
From frozen yoghurt results it could be concluded that, the acidity was increased in treatment 2 than treatment 1 and this may be due to the presence of sorbitol. Specific gravity of the mix as well as the resultant low calorie yoghurt were decreased in the presence of sorbitol. Weight per gallon was closely related to specific gravity values. Freezing point decreased slightly in treatment 2 than treatment 1. Overrun percent increased in treatment 2 than treatment 1. But the differences between the treatments were not significant. Melting resistance for treatment 2 was low than treatment 1. The difference between treatments in organoleptic properties were not significant.

Finally, this study recommended that an acceptable and palatable frozen yoghurt can be made by using sorbitol as a partial substitute of sucrose.

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STUDIES ON LOW CALORIE ICE MILK

1- THE USE OF SUCROL AND SORBITOL

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Low calorie ice milk was made by using different percentages of sucrol and sorbitol as a partial substitute of sucrose. The formula used for making the low calorie ice milk contained 2% fat, 15% sucrose, 13% milk solid not fat milk and 0.7% gelatin. The low calorie ice milk mix was analysed for acidity, specific gravity, weight per gallon, viscosity and freezing point. The resultant low calorie ice milk was analyzed for specific gravity, weight per gallon, overrun, melting resistance, organoleptic evaluation, and energy content. It was concluded that sucrol and sorbitol can be used as a partial substitute for sucrose (2% sucrol or sorbitol, 1% sucrol or sorbitol and 1% sorbitol + 1% sucrol) to produce an acceptable and palatable ice milk.

Obesity is one of the most common nutritional disorders in present days especially in development and fast developing countries (1), and it represents a serious health hazards. Excessive weight increase susceptibility to a number of diseases, such as diabetes mellitus, hypertension, renal diseases, arteriosclerosis, cardiovascular diseases, gall bladder, renal diseases and others. Low fat ice milk is suitable to obese people and to those who suffer

from arteriosclerosis and cardiovascular diseases. Low calorie ice milk is characterized by: low fat, low cholesterol, low sugar (sucrose). In addition to its content of protein with high biological value, it contains calcium, carbohydrate and vitamins. Sommer (2) stated that the proteins in ice cream are high, and completely assimilated than most other proteins. Ice cream is an easily digestible and highly palatable food. It possesses special advantages as a food for various types of patients and convalescents.

In this study commercial sweetening agents sucrol and sorbitol were used as a partial substitute for sucrose in making ice milk.

Material and methods:

Fresh cow's milk used in this study was obtained from the herd of Faculty of Agriculture, Minufiya University, Egypt. Skim milk powder was an U.S.A. low heat product. Gelatine, vanilla, sucrol and sucrose were Egyptian products. Sorbitol was a brand called, sorbitol Neosorb Requite P11 420. Stabilizer used in this study was Palsgaard (emulsifier and stabilizer) produced by Palsgaard Industrie A/S, Denmark.

Preparation of low calorie ice milk:

Low calorie ice milk using sucrol and sorbitol as a partial substitute for sucrose was done as follow:

It was made from milk standardized to 2% fat and 13% solid-not-fat and 0.7% gelatine, suitable amount of vanilla and different amount of sugars as follow:

Treatment No. 1 contained 15% sucrose (as a control). In each batch approximately 1kg of the mix was consisted of 830 gm, standardized milk, 33.2gm. skim milk powder, 129.5gm. sucrose and 6.95gm. gelatine.

Treatment No. 2 contained 14% sucrose and 1% sucrol. In each batch approximately 1kg of the mix was consisted of 830 gm standardized milk, 33.2gm skim milk powder, 120.87gm sucrose, 8.63gm sucrol and 6.95gm gelatine.

Treatment No. 3 contained 13% sucrose and 2% sucrol. In each batch approximately 1kg of the mix consisted of 830gm standardized milk, 33.2gm skim milk powder, 112.24gm sucrose, 17.26gm sucrol and 6.95gm gelatine.

Treatment No. 4 contained 14% sucrose and 1% sorbitol. In each batch approximately 1kg of the mix

consisted of 830 gm standardized milk, 33.2gm skim milk powder, 120.07gm sucrose, 8.63gm sorbitol and 6.95gm gelatine.

Treatment No. 5 contained 13% sucrose and 2% sorbitol. In each batch, approximately 1kg of the mix consisted of 830gm standardized milk, 33.2gm skim milk powder, 112.24gm sucrose, 17.26gm sorbitol and 6.95gm gelatine.

Treatment No. 6 contained 13% sucrose, 1% sucrol and 1% sorbitol.

In each batch, approximately 1kg of the mix consisted of 830gm standardized milk, 33.2gm skim milk powder, 112.24gm sucrose, 8.63gm sucrol, 8.36gm sorbitol and 6.95gm gelatine.

Methods of analysis:

a- Mix properties:

Acidity was determined according to Henry et al. (3).

Specific gravity was determined according to Winton (4).

Weight per gallon of the mix was directly calculated according to Burke (5) and Arbuckle (6).

Viscosity was carried out by using Hoppler viscometer type BH₂ No. 17663 with calibrated tube No. 69016.

Freezing point was determined according to the method recommended for milk by the FAO. Regional Dairy Development and Training Center for the Near East (7).

b- Analytical methods for the resultant ice milk:

Specific gravity and weight per gallon were determined.

Overrun of the frozen product was calculated using the following equation (8).

$$\% \text{ overrun} = \frac{\text{Weight of mix-weight of frozen ice cream}}{\text{Weight of frozen ice cream}} \times 100$$

Melting resistance was determined according to Reid and Painter (9).

Organoleptic scoring was carried out by regular panel from the Food Science Department, Faculty of Agriculture. Scoring was carried out according to Rothwell (10), for flavour (50 points), body and texture (40 points) and melting quality (10 points).

The caloric value of 100gm of ice milk was calculated according to Arbuckle (6) and Mottram (12).

The data were statistically analyzed (ii).

Results and discussion:

a- Mix properties:

- 1- Acidity: It was ranged from 0.17 to 0.19% (Table 1) and the analysis of variance showed that the differences between the treatments were not significant.
- 2- Specific gravity and weight per gallon: Partial replacement of sucrose with sucrol and sorbitol showed that the mean values of specific gravity and weight per gallon ranged from 1.1016 to 1.1553 and from 4.17 to 4.37kg respectively (Table 1). Analysis of variance showed that the differences for both were not significant.
- 3- Freezing point: The mean values of freezing point ranged from -2.14 to -2.32°C (Table 1). However, the freezing point decreased by increasing the amount of sucrol and sorbitol added. The freezing point of the mix depends on the soluble constituents in the mix and their molecular weight. Soluble constituents with high molecular weights will cause the least lowering of freezing point, and lower molecular weight constituents will depress the freezing point to a greater extent (13). Our results agreed with the observation of Bae and Bladwin (14) who found that sorbitol caused greater freezing

Table (1) : Effect of partial replacement of sucrose with sucral and sorbitol on acidity, specific gravity, weight per gallon (Wp) and freezing point of low calorie ice milk mix.

Treatment No.	Sucrose %	Sorbitol %	Sucral %	Acidity %	Specific gravity	Weight per gallon (Wp)	Freezing point (C)
(Control) 1	15	-	-	0.17	1.1230	4.29	-2.14
2	14	-	1	0.19	1.1353	4.30	-2.23
3	13	-	2	0.18	1.1456	4.31	-2.28
4	14	1	-	0.17	1.1378	4.31	-2.30
5	13	2	-	0.18	1.1016	4.17	-2.32
6	13	1	1	0.19	1.1553	4.37	-2.32

Table (2): Effect of partial replacement of sucrose with sucral and sorbitol on the viscosity (Cp) of low calorie ice milk mixes aged for different periods.

Treatments No.	Sucrose %	Sorbitol %	Sucral %	Viscosity (Cp) of mix aged for :		
				0.0hour	4 hours	24 hours
Control 1	15	-	-	5.19	10.99	127.07
2	14	-	1	5.85	14.80	126.50
3	13	-	2	6.42	21.81	136.90
4	14	1	-	8.51	24.25	136.65
5	13	2	-	5.13	23.19	129.00
6	13	1	1	4.79	27.24	134.49

point depression than sucrose. Analysis of variance showed that the differences between the treatment were not significant.

- 4- Viscosity: Table (2) shows the viscosity of low calorie ice milk mix aged for different periods. The increase in the viscosity was slight in the fresh mixes, and high in the 24 hours aged samples. This sharp increase in the viscosity after 24 hours of aging could be due to the hydration process of initial milk protein and to the effect of the stabilizer used (13). Analysis of variance showed that the differences between treatments were significant after 4 hours of aging.

b- Low calorie ice milk properties:

- 1- Specific gravity and weight per gallon. As shown in Table 3, the weight per gallon was closely related to the specific gravity. Specific gravity and weight per gallon of low calorie ice milk were decreased when 1% of sucrol or sorbitol was used and increased when the amount of sucrol or sorbitol added increased. Statistical analysis proved that the changes in both specific gravity and weight per gallon were not significant.

Table (3): Effect of partial replacement of sucrose with sucrol and sorbitol on specific gravity, weight per gallon and overrun of low caloric ice milk.

Treatments No.	Sucrose	Sorbitol	Sucrol	Specific gravity	Weight per gallon (Kg)	Overrun %
	%	%	%			
Control (1)	15	-	-	0.7386	2.80	62.67
(2)	14	-	1	0.7285	2.76	55.72
(3)	13	-	2	0.7397	2.80	60.25
(4)	14	1	-	0.7130	2.70	66.15
(5)	13	2	-	0.7297	2.76	55.43
(6)	13	1	1	0.7108	2.69	55.37

Table (4): Effects of partial replacement of sucrose with sucrol and sorbitol on the melting resistance of low caloric ice milk.

Treatments No.	Sucrose %	Sorbitol %	Sucrol %	Loss Percentage after		
				First 60 minutes	Next 30 minutes	Last 30 minutes
Control (1)	15	-	-	7.47	24.00	69.45
(2)	14	-	1	4.48	20.17	75.56
(3)	13	-	2	8.90	36.33	54.76
(4)	14	1	-	10.62	26.58	61.81
(5)	13	2	-	16.62	26.39	63.10
(6)	13	1	1	6.25	16.04	78.79

- 2- Overrun: Table 3 shows that the overrun decreased in all treatments, except treatment No. 4, the overrun increased, and this may be due to its low specific gravity.
- 3- Melting resistance: Melting resistance means the percent loss in weight of the initial weight of the tested sample. Table 4 shows that low calorie ice milk had a tendency to be low resistant as sucrol and sorbitol content decreased. This is may be due to the effect of sucrol and sorbitol on the freezing point of the mix. The statistical analysis showed that the differences between treatments were significant.
- 4- Organoleptic evaluation:
- a- Low calorie ice milk using 1% sucrol and 14% sucrose. Table 5 shows that treatment 1 had a higher flavour score than treatments 2 and 3. The decrease in the flavour score of treatments 2 and 3 may be due to the unlike after taste of sucrol. Also the results indicated that the score averages of body and texture were 32.8, 31.2 and 30.7 points for treatments 1, 2 and 3 respectively. From the same table it is clear that the melting quality decreased when sucrol was added, and there was a slight decrease in

Table (5): Effect of sucral (1%) with sucrose (14%) on the organoleptic properties of low caloric ice milk.

Treatments No.	Sucrose %	Sucral %	Flavour (50 points)	Body & texture (40 points)	Melting quality (10 points)	Total score (100 points)
Control(1)	15	—	41.7	32.8	8.0	82.5
(2)	14	1	40.8	31.2	7.0	77.3
(3)	14	1	38.5	30.2	7.7	76.7

Table (6) : Effect of sucral (2%) with 13% sucrose on the organoleptic properties of low caloric ice milk.

Treatments No	Sucrose %	Sucral %	Flavour (50 points)	Body & texture (40 points)	Melting quality (10 points)	Total score (100 points)
Control(1)	15	—	42.2	36.5	8.8	85.8
(2)	13	2	42.2	30.3	6.3	80.5
(3)	13	2	42.3	30.8	6.3	79.5

the total score of treatments 2, 3 than treatment (1), control sample. Analysis of variance indicated that the effect of using 1% sucrol with 14% sucrose on the organoleptic properties was not significant.

b- Low calorie ice milk using 2% sucrol and 13% sucrose. Results given in Table 6 indicate that melting quality decreased in treatments 2 and 3 than treatment 1. This decrease may be due to the effect of sucrol on the freezing point of low calorie ice milk mix. Also there was a slight decrease in the total score of treatments 2 and 3 than treatment 1. The analysis of variance showed that the processing of low calorie ice milk using 2% sucrol with 13% sucrose had no significant effect on the organoleptic properties.

c- Low calorie ice milk using 1% sorbitol and 14% sucrose. Table 7 shows that treatment 1 had the highest score for both flavour and body and texture. Melting quality decreased in treatments 2 and 3 than treatment 1. Also, there was a slight increase in the total score of treatment 1 than treatments 2 and 3. The analysis of variance showed that the changes occurred in the total score were significant.

Table (7) : Effect of sorbitol 1% with sucrose 14% on the organoleptic properties of low calorie ice milk.

Treatments	Sucros	Sorbitol	Flavour	Body & Texture	Melting quality	Total score
No	%	%	(50 points)	(10 points)	(10 points)	(100 points)
Control (1)	15	-	43.4	37.5	9.3	90.2
(2)	14	1	37.8	31.8	8.3	81.0
(3)	14	1	38.4	31.3	8.2	80.8

Table (8) : Effects of sorbitol 2% with 13% sucrose on the organoleptic properties of low calorie ice milk.

Treatments	Sucrose	Sorbitol	Flavour	Body & Texture	Melting quality	Total score
No.	%	%	(50 points)	(50 points)	(10 points)	(100 points)
Control (1)	15	-	42.5	33.8	7.7	84.0
(2)	13	2	41.2	32.0	7.2	80.3
(3)	13	2	41.6	32.5	7.2	81.2

d- Low calorie ice milk using 2% sorbitol and 13% sucrose. Table 8 indicates that treatment 1 had a higher flavour score than treatments 2 and 3. The decrease in the flavour score of treatments 2 and 3 may be due to the presence of sorbitol (2%), since sorbitol is a one half of sucrose as a sweetener (15). Analysis of variance showed no significant difference between treatments.

e- Low calorie ice milk using 1% sucrol, 1% sorbitol and 13% sucrose: Results given in Table 9 indicated that treatment 1 had the highest score for flavour, body and texture, melting quality, and total score, but the differences were not significant.

Energy content: Data given in Table 10 indicate that the reduction of the energy value was 37.37% for treatment 1 when compared with the standard treatment which contained 15% sucrose, 12.5% fat, 11.5% milk solids non fat and 0.3% gelatin (6). The reduction of energy value was 4.0% for all treatments when compared with treatment 1 which contained 15% sucrose, 3% fat, 12% solid non fat, 0.0% gelatin (16).

Table (9) : Effect of sucral 1% + sorbitol 1% with sucrose 13% on the organoleptic properties of low calorie ice milk.

Treatments No.	Sucrose %	Sorbitol %	Sucral %	Flavour (30 points)	Body & texture (40 points)	Melting quality (10 points)	Total score (100 points)
Control (1)	15	-	-	41.2	34.2	7.8	83.2
(2)	13	1	1	40.7	31.5	7.3	82.5
(3)	13	1	1	39.3	32.2	7.7	79.2

Table (10) : Effect of sucral and sorbitol replacement part on the energy content of low calorie ice milk. [Kilo Joule / 100gm].

Treatments	Sucrose %	Sorbitol %	Sucral %	Kilo Joule / 100 gm	Reduction of energy %
Standard treatment	15	-	-	870.44	-
Treatment (1)	15	-	-	545.20	37.37
(2)	15	-	-	519.04	4.80
(3)	14	-	1	519.04	4.80
(4)	13	-	2	519.04	4.80
(5)	14	1	-	519.04	4.80
(6)	13	2	-	519.04	4.80
(7)	13	1	1	519.04	4.80

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Studies on Low Calorie Ice Milk

2- The use of corn oil

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Experiments were conducted to study the effect of adding different levels of corn oil on some properties of low calorie ice milk mixes (2% fat, 13% milk solids non fat and 0.7% gelatin) (control or treatment 1) as well as the resultant low calorie ice milk. Increasing added corn oil decreased the acidity in all treatments except treatment which contains 2% sorbitol + 2% sucrose + 2% corn oil. Specific gravity, weight per gallon, freezing point and overrun were decreased when the amount of corn oil used increased. Melting resistance of low calorie ice milk treatments were correlated with the freezing point. Using 1% corn oil with 1% cow's milk fat + 15% sucrose or using 1% corn oil + 1% cow's milk fat + 13% sucrose + 2% sorbitol produced low calorie ice milk with similar quality as compared with the sample which contained 2% cow's milk fat + 15% sucrose (control).

INTRODUCTION

The use of vegetable fats and oils instead of butter fat in the manufacture of ice cream is of interest for various reasons, such as price, consistency, whippability, stability and nutrition. Hausmen⁽¹⁾ carried out a survey on the production of ice cream with peanut butter flavour at eight manufactures in the U.S.A. This survey showed that most companies used peanut butter in their ice cream recently have obtained commercial success. Madsen⁽²⁾ reported that vegetable fats and oils in ice cream improved whippability, stability and the emulsifier component also has a significant influence. This study was carried to study the effect of adding different levels of corn oil in making low calorie ice milk.

Material and Methods

Fresh cow's milk was obtained from the herd of Faculty of Agriculture Minufiya University. Skim-milk powder was an U.S.A. low heat product. Sucrose was a crystalline commercial grade produced by the Egyptian sugar and Distillation Company. Corn oil was a commercial brand "Coroli" produced by Cebag B-V-Zwolle-Holland. Sorbitol used in this study was a brand called sorbitol Neosorb Requite pll 450. Palsgaard 5936 (emulsifier and stabilizer), produced by palsgaard industrie A/S, Denmark, was used. Vanilla and gelatin were obtained from the local market.

Low calorie ice milk was made by using corn oil as a partial substitute of cow's milk fat. It was prepared as follow:

Treatment 1 (control)	2% cow's milk fat + 15% sucrose.
Treatment 2	0.5% corn oil + 1.5% cow's milk fat + 15% sucrose.
Treatment 3	1% corn oil + 1% cow's milk fat + 15% sucrose.
Treatment 4	2% corn oil + 15% sucrose.
Treatment 5	0.5% corn oil + 1.5% cow's milk fat + 13% sucrose + 2% sorbitol.
Treatment 6	1% corn oil + 1% cow's milk fat + 13% sucrose + 2% sorbitol.
Treatment 7	2% corn oil + 13% sucrose + 2% sorbitol.

Method of Manufacture:

The mix was sifted slowly to the standardized milk at 45°C under vigorous agitation to prevent lumping, and the required amount of palsgaard (0.7%) was added after dissolving in warm water. The mix was pasteurized at 72°C for 30 minutes, then cooled to 5°C and aged at that temperature for 24 hours prior to freezing (6). After aging, a suitable amount of vanilla was added, then each batch was frozen in an experimental ice cream freezing machine (Kaprice-Cattabriga). This freezer was auto-

matically controlled to stop whipping when ice cream reaches the right consistency. It was packed in cups 100 gm., and hardened in a cabinet held at -25°C for 24 hours.

Methods of Analysis:

- a- Mix Properties: The mix was examined for acidity⁽³⁾, specific gravity⁽⁴⁾, weight per gallon (5 & 6), viscosity using viscometer type BH₂ No. 17663 with calibrated glass tube No. 69016 and freezing point according to the method recommended for milk by the FAO Regional Dairy Development and Training Center for the Near East⁽⁷⁾.
- b- Resultant ice milk: The resultant ice milk was studied for specific gravity⁽⁴⁾, weight per gallon^(5,6), overrun⁽⁸⁾, melting resistance⁽⁹⁾ and organoleptic evaluation⁽¹⁰⁾.

The organoleptic evaluation was carried out by regular panel from the Food Science Department, Faculty of Agriculture, Minufiya University. Evaluation was carried according to Rothwell⁽¹⁰⁾, for flavour (50 points), body and texture (40 points) and 10 points for melting quantity.

- c- Statistical Analysis: The data were subjected to analysis of variance⁽¹¹⁾.

Results and Discussion

a- Mix Properties:

1- Acidity: Table (1) shows that the acidity decreased in treatments 2,3,4 and 6 while it increased slightly in treatment 7. Analysis of various indicated that there were no significant differences between treatments.

2- Specific gravity and weight per gallon: They decreased when the level of corn oil increased (Table 1). The differences between treatments were significant.

3- Freezing point: As shown in table (1) the freezing point was slightly decreased when the level of corn oil increased. The differences between treatments were significant.

4- Viscosity: It was very low in treatment 1. This is may be due to the presence of gelatin as a stabilizer in this treatment, but in treatments 2 to 7, Palsgaard was used as a stabilizer emulsifier to corn oil. Also viscosity decreased when the amount of corn oil increased (Table 1). The differences between treatments were insignificant.

b- Resultant ice milk properties:

1- Specific gravity and weight per gallon: Table (2) shows the effect of different percentages of corn

Table (1). Effect of partial replacement of cow's milk fat with corn oil on the acidity, specific gravity, weight per gallon, freezing point and viscosity (after 4 hours) of low calorie ice milk mixes.

Treatments	Com oil %	Cow's milk % fat	Sucrose %	Sorbitol %	Acidity %	Specific Gravity	Weight per gallon (Kg)	Freezing point °C	Viscosity after 4 hours (Cp)
Control (1)	-	2	15	-	0.17	1.1303	4.28	-2.09	10.97
(2)	0.5	1.5	15	-	0.14	1.1013	4.17	-2.10	54.57
(3)	1	1	15	-	0.16	1.0800	4.09	-2.11	51.90
(4)	2	-	15	-	0.16	1.0500	3.98	-2.12	49.24
(5)	0.5	1.5	13	2	0.17	1.0800	4.09	-2.16	56.27
(6)	1	1	13	2	0.16	1.0733	4.06	-2.30	54.55
(7)	2	-	13	2	0.19	1.0400	3.94	-2.85	53.28

oil on specific gravity, weight per gallon and overrun of low calorie ice milk. From this table it was clear that the specific gravity decreased when the percentage of corn oil increased. The changes in specific gravity may be due to the differences between cow's milk fat and corn oil in unsaturated fatty acids content. Weight per gallon was proportional to specific gravity value in all treatments. The differences between treatments for specific gravity and weight per gallon were not significant.

- 2- Overrun: Results given in table (2) indicate that the overrun percentage of low calorie ice milk decreased as the amount of corn oil increased or when the specific gravity decreased. The decrease in the overrun might be due to the decreases in the viscosity as the level of corn oil increased. These results were in agreement with those of Salama⁽¹⁴⁾ who found that the overrun decreased as the viscosity of the mix decreased. Also these results agreed with those of Hofi⁽¹⁵⁾ who mentioned that when the specific gravity of the mix decreased the overrun percentage also decreased. Analysis of variance indicated that there was significant difference between treatments.

Table (2) : Effect of partial replacement of cow's milk fat with corn oil on specific gravity, weight per gallon and overrun of low caloric ice milk.

Treatments No.	Corn oil %	Cow's milk fat %	Sucrose %	Sorbitol %	Specific gravity	Weight per gallon (Kg)	Overrun %
Control (1)	-	2	15	-	0.6936	2.63	65.46
(2)	0.5	1.5	15	-	0.6837	2.59	64.50
(3)	1	1	15	-	0.6766	2.56	63.70
(4)	2	-	15	-	0.6738	2.55	60.03
(5)	5	1.5	13	2	0.6779	2.57	63.00
(6)	1	1	13	2	0.6668	2.52	61.39
(7)	2	-	13	2	0.6400	2.42	53.12

Table (3) : Effect of partial replacement of cow's milk fat with corn oil on melting resistance of low caloric ice milk

Treatments No.	Corn Oil %	Cow's milk fat %	Sucrose %	Sorbitol %	Loss percent after :		
					First 60 minutes	Next 30 minutes	Last 30 minutes
Control (1)	-	2	15	-	7.39	24.38	58.83
(2)	0.5	1.5	15	-	12.50	34.40	48.56
(3)	1	1	15	-	13.20	37.40	42.30
(4)	2	-	15	-	16.51	41.50	38.50
(5)	0.5	1.5	13	2	16.25	41.63	32.90
(6)	1	1	13	2	21.15	43.03	26.90
(7)	2	-	13	2	31.10	48.20	11.90

3- Melting resistance: At the first hour of exposure to 30°C, the low calorie ice milk treatments had a high resistance. While treatment 1 lost 7.39% of its weight after one hour, the corresponding values were 12.5%, 13.20%, 16.51%, 16.25%, 21.15% and 31.10% for treatments 1 to 7, respectively table (3). This decrease in melting resistance may be due to increases in viscosity. These results agreed with those of Hofi et. al.(16) who found that the melting resistance of the resulting ice cream decreased when the viscosity of the mix increased. During the next 30 minutes and the last 30 minutes, the different low calorie ice milk samples showed the same previous trend which occurred during the first period. From table (3) it could be concluded that the highest resistance was obtained with treatment 7 which contained 2% corn oil + 13% sucrose + 2% sorbitol. The differences between treatments were significant.

4- Organoleptic evaluation: Organoleptic properties of the resultant low calorie ice milk as affected by corn oil are shown in Table (4). From this table it was clear that using 1% corn oil with 1% cow's milk fat produced low calorie ice milk with similar quality as compared with treatment 1 (2% cow's milk fat). Analysis of variance, showed that the difference between treatments were significant.

Table (4) : Effect of partial replacement of cow's milk fat with com oil on organoleptic properties of low calorie ice milk.

Treatments	Com oil	Cow's milk fat	Sucrose	Sorbitol	Flavour	Body and texture	Melting quality	Total score
No.	%	%	%	%	(50 points)	(40 points)	(10 points)	(100 points)
Control (1)	-	2	15	-	45.2	36.7	8.8	90.7
(2)	0.5	1.5	15	-	44.2	32.7	8.3	85.2
(3)	1	1	15	-	43.2	35.0	8.0	86.2
(4)	2	-	15	-	39.2	30.2	6.3	75.3
(5)	0.5	1.5	13	2	44.5	32.5	7.2	84.2
(6)	1	1	13	2	43.8	34.3	7.3	85.5
(7)	2	-	13	2	39.0	31.0	6.3	76.3

In conclusion, mix containing 1% corn oil, 1% cow's milk fat and 15% sucrose or 1% corn oil, 1% cow's milk fat, 13% sucrose and 2% sorbitol produced low calorie ice milk with similar quality as treatment I which contained 2% cow's milk fat and 15% sucrose (control).

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Studies on Low Calorie Ice Milk

3- The use of strawberry and apricot

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ABSTRACT

This study was conducted to study the effect of using strawberry and apricot fruits as a partial substitute for sucrose on some properties of the ice milk mixes as well as the final product. These properties were: acidity, specific gravity, weight per gallon, freezing point, melting resistance, organoleptic properties and energy content. It was found that acidity of the mixes increased while specific gravity, weight per gallon and freezing point decreased when fruits were added. Also specific gravity and weight per gallon of the final product decreased while overrun was slightly increased. Also energy content was decreased. So using strawberry or apricot fruits in making ice milk gave a more acceptable and palatable product.

INTRODUCTION

The ice milk is one of the chief markets for fresh, frozen and canned fruits. Fabricius (1) carried a great deal of experiments on the use of strawberries for ice cream manufacture. His experiments proved that cold-packed strawberries were superior to canned berries and that both gave products that were superior to those

packed using strawberry extracts for flavouring. Bray (2) found that the use of tropical fruit in the processing of ice cream improved its nutritive value. In the present study strawberry and apricot fruits were used as a partial substitution for sugar (sucrose) and as flavouring agent in ice milk. These fruits were added in juice form before whipping.

Material and Methods

Ingredients are the same as in part one of this series (3). In addition low calorie fruits, strawberry or apricots were used. These fruits were purchased from the local market.

Methods:

Preparation of fruit juice: Strawberry or apricot was washed, cut into small pieces and homogenized very well in a food processing machine (Blender). The fruit juice was heated separately in a water bath at 80°C for 15 minutes. Total solid was determined by using Abbe Refractometer to calculate the amount of fruit juice which substitutes the amount of sugars.

Preparation of low calorie ice milk: Low calorie ice milk was prepared as follow:

Group A:

- Treatment no. 1 (control). contained 15% sucrose and suitable amount of vanilla.
- Treatment no. 2 contained 13.5% sucrose + 1.5% strawberry solids (30 gm strawberry juice).

Each batch weighted approximatly (1 Kg) consisted of 830 gm standardized milk 33.2 gm skim milk powder, 116.532 gm sucrose, 12.95 gm strawberry solid [258.96 gm strawberry juice] and 6.95 gm gelatine.

- Treatment no. 3 contained 11.5% sucrose, 2% sorbitol and 1.5% strawberry solids (30 gm strawberry juice).

Each batch weighted approximatly (1 Kg) consisted of 830 gm standardized milk, 33.2 gm skim milk powder, 99.268 gm sucrose, 17.26 gm sorbitol, 12.95 gm strawberry solid (258.96 gm strawberry juice) and 6.95 gm gelatine.

Group B:

- Treatment no. 1 (control) contained 15% sucrose + suitable amount of vanilla.
- Treatment no. 2 contained 13% sucrose + 2% apricot solids [25 gm natural apricot juice].

Each batch weighted approximatly (1 Kg) consisted of 830 gm standardized milk, 33.2 gm skim milk powder, 112.22 gm sucrose, 17.26 gm apricot solid (215.8 gm apricot juice) and 6.95 gm gelatine.

- Treatment no. 3 contained 11% sucrose, 2% sorbitol
2% apricot solids [25 gm natural apricot juice].

Each batch weighted approximately (1 Kg) consisted of 830 gm standardized milk, 33.2 gm, skim milk powder, 94.95 gm sucrose, 17.26 gm sorbitol, 17.26 gm apricot solid (215.8 gm apricot juice) and 6.95 gm gelatine.

a- Mix properties: The following properties were determined: acidity (4), specific gravity (5), weight per gallon (6,7), viscosity and freezing point (8).

b- Resultant ice milk properties: The resultant low calorie ice milk was studied for specific gravity (5), weight per gallon (6), overrun (9), melting resistance (10), organoleptic scoring (11) and energy content (7,12).

The data were subjected to analysis of variance (13).

Results and Discussion

Group A: (Strawberry low calorie ice milk):

a- Mix properties:

1- Acidity: Table (1) shows the effect of strawberry and sorbitol on the acidity, specific gravity, weight per gallon and freezing point of low calorie ice milk mixes. There was a considerable increase in the acidity

of treatments 2 and 3 than treatment 1 (control). This increase might be due to the acid nature of strawberry. Analysis of variance showed that the differences between treatments 1,2 and 3 were significant. On the other hand the differences between treatments 2 and 3 were not significant.

2- Specific gravity and weight per gallon: It is noticed that the specific gravity decreased in the presence of strawberry (Table 1). This decrease is due to the presence of fructose in strawberry with a low molecular weight = 180, while the molecular weight of sucrose = 342. So the presence of fructose in treatments 2 and fructose with sorbitol (low molecular weight) in treatment 3, are the cause of decreasing the specific gravity in both treatments than treatment 1 (7,14). Weight per gallon of the mixes were closely related to the specific gravity of the corresponding mixes. Analysis of variance indicated that there was a significant difference between treatments.

3- Freezing point: The freezing point decreased in treatments 2 and 3 Table (1) and this might be due to the presence of fructose in strawberry and sorbitol, a low molecular weight sugars (7,14). Analysis of variance showed that the differences between treatments were significant.

Table (1) : Effect of using low energy fruit [Strawberry] and sorbitol on the acidity, specific gravity, weight per gallon and freezing point of low calorie ice milk mixes.

Treatments No.	Strawberry solids %	Sucrose %	Sorbitol %	Acidity %	Specific gravity	Weight per gallon(Kg)	Freezing point °C
Control (1)	—	15	—	0.19	1.1263	4.26	-2.15
(2)	1.5	13.5	—	0.53	1.0844	4.11	-3.01
(3)	1.5	11.5	2	0.58	1.0607	4.01	-3.11

Table (2) : Effect of adding strawberry fruit on the specific gravity, weight per gallon and overrun of low calorie ice milk.

Treatments No.	Strawberry solids %	Sucrose %	Sorbitol %	Specific gravity	Weight per gallon (Kg)	Overrun %
Control (1)	—	15	—	0.7554	2.86	59.58
(2)	1.5	13.5	—	0.7535	2.85	59.72
(3)	1.5	11.5	2	0.6641	2.51	63.08

b- Resultant strawberry low calorie ice milk properties:

1- Specific gravity and weight per gallon. Table (2) showed that the specific gravity for treatment, 1 was 0.7554 then decreased from 0.7535 to 0.6641 for treatments 2 and 3 respectively. Weight per gallon was proportional to the specific gravity in all treatments. Analysis of variance showed that the differences between treatments were significant.

2- Overrun: Results given in Table (2) indicated that the overrun value was slightly increased in treatments 2 and 3 than treatment 1. This increase might be due to the decrease in the specific gravity and these results are in agreement with those of Hofi et al (15), who found that the overrun percentage increased when the specific gravity decreased. Analysis of variance was not significant.

3- Melting resistance: Table (3) indicated that the lowest resistance was obtained with treatment 1 which contained 15% sucrose. Differences in melting property are mainly due to the differences in the freezing point of the mixes. These results are in agreement with those of Hofi (16). There were no significant differences between treatments.

Table (3) : Effect of adding strawberry fruit on the melting resistance of low calorie ice milk.

Treatments	Strawberry solids	Sucrose	Sorbitol	Loss percent after :-		
				First 60 minutes	Next 30 minutes	Last 30 minutes
No.	%	%	%			
Control (1)	--	15	--	8.23	24.67	60.00
(2)	1.5	13.5	--	23.73	38.03	26.20
(3)	1.5	11.5	2	25.39	43.45	24.16

Table (4) : Effect of adding strawberry fruit on the organoleptic properties of low calorie ice milk.

Treatments	Strawberry solids %	Sucrose %	Sorbitol %	Flavour (50 points)	Body and texture (40 points)	Melting quality (10 points)	Total score (100 points)
No.							
Control (1)	--	15	--	36.7	36.5	8.5	81.7
(2)	1.5	13.5	--	35.1	34.3	8.3	78.3
(3)	1.5	11.5	2	35.3	35.0	8.3	78.7

4- Organoleptic properties: Table (4) showed that the score averages for flavour were slightly decreased as the strawberry was added. Also there was a decrease in body and texture when adding strawberry and this decrease might be due to increase in acidity percent. When acidity percentage increases in the mix, the ice crystal size increases, thus produces a coarse texture (7). Also this table showed that the melting quality was decreased in treatment 1 and this might be due to the effect of fructose on the freezing point of the mixes. Analysis of variance for total score points indicated that the effect of strawberry, sucrose and sorbitol was not significant.

5- Energy content: It was noticed that table (5), the reduction of energy value was 37.37% for treatment 1 when compared with the standard treatment which contained 15% sucrose, 12.5% fat, 11.5% milk solid non fat, and 0.3% gelatin (7), and the reduction of energy was 39.7% for treatments 2 and 3 when also compared with the standard treatment which contained 15% sucrose, 3% fat, 13% milk solid non fat and 0.8% gelatin (17).

Group B (Apricot low calorie ice milk):

a- Mix properties:

Acidity, specific gravity, weight per gallon and

Table (5) : Effect of using low energy fruit [strawberry] on the energy content of low caloric ice milk. (Kilo Joule / 100 gm).

Treatments No.	Strawberry solids %	Sucros %	Sorbitol %	Kilo Joule / 100 gm	Reduction of energy % than the standard
Standard treatment	--	15	--	870.44	--
Treatment (1)	--	15	--	545.20	37.37
(2)	1.5	13.5	--	524.85	39.7
3.73 (3)	1.5	11.5	2	524.85	39.7

Table (6) : Effect of using low energy fruit [apricot] on the acidity, specific gravity, weight per gallon and freezing point of low caloric ice milk mixes.

Treatments No.	Apricot solids %	Sucrose %	Sorbitol %	Acidity %	Specific gravity	Weight per gallon (Kg)	Freezing point C
Control (1)	--	15	--	0.17	1.1301	4.28	-2.14
(2)	2	13	--	0.76	1.1226	4.52	-3.16
(3)	2	11	2	0.96	1.0809	4.09	-3.18

Table (7) : Effect of adding apricot fruit on the specific gravity, weight per gallon and overrun of low calorie ice milk.

Treatments No.	Apricot solids %	Sucrose %	Sorbitol %	Specific gravity	Weight per gallon (Kg)	Overrun %
Control (1)	--	15	--	0.7121	2.70	65.23
(2)	2	13	--	0.6461	2.45	66.37
(3)	2	11	2	0.6445	2.44	68.19

Table (8) : Effect of adding apricot fruit on the melting resistance of low calorie ice milk.

Treatments No.	Apricot solids %	Sucrose %	Sorbitol %	Loss percent after		
				First 60 minutes	Next 30 minutes	Last 30 minutes
Control (1)	--	15	--	7.25	32.80	59.00
(2)	2	13	--	25.39	42.43	25.33
(3)	2	11	2	30.02	44.30	16.82

Table (10) : Analysis of variance for melting resistance of apricot low calorie ice milk.

Table (9) : Effect of adding apricot fruit on the organoleptic properties of low caloric ice milk.

Treatments No.	Apricot solids %	Sucrose %	Sorbitol %	Flavour (50 points)	Body and texture (10 points)	Melting quality (10 points)	Total score (100 points)
Control (1)	--	15	--	41.8	36.0	8.3	85.3
(2)	2	13	--	41.7	32.2	6.8	80.6
(3)	2	11	2	40.2	33.2	7.5	80.8

Table (10) : Effect of using low energy fruit (Apricot) on the energy content of low caloric ice milk (Kilo Joule / 100 gm).

Treatments No.	Apricot Solids %	Sucrose %	Sorbitol %	Kilojoule / 100 gm	Reduction of energy Than the standard %
Standard treatment	--	15	--	870.44	--
Treatment (1)	--	15	--	545.20	37.37
(2)	2	13	--	521.75	40.0
(3)	2	11	2	521.75	40.0

freezing point had the same results as that obtained in case of using strawberry (Table 6).

b- Resultant apricot low calorie ice milk properties:

The results obtained in case of using apricot in making low calorie ice milk are the same as in case of using strawberry (Table 7, 8, 9, 10).

It was concluded that low calorie ice milk could be made successfully with strawberry or apricot and sucrose or strawberry or apricot with sucrose and sorbitol without any significant differences than low calorie ice milk with 15% sucrose.

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***Comparative Biological Effects Of Green S And Chlorophyll
Feeding In Albino Rats.***

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Abstract

The effects of feeding Chlorophyll and Green S on carbohydrate, lipid and protein metabolism were studied. Body weight gain, liver weight and liver glycogen were significantly decreased, but blood glucose was markedly elevated as compared to controls. Total lipids, total cholesterol, phospholipids and triacylglycerols were slightly decreased. Green S feeding resulted in significant decrease of total lipids as compared to controls and to rats fed Chlorophyll.

Aminotransaminases were slightly stimulated, while lactate dehydrogenase was markedly inhibited on feeding food colourants. Serum total proteins and gamma-globulin were significantly elevated, while albumin, alpha1-, alpha2- and beta-globulins showed slight variations as compared to controls.

Our histological results were mainly found with rats receiving Green S. The inflammatory foci seen in the hepatic parenchyma showed higher incidence in rats fed with the synthetic dyes, which might be due to the toxic effect of Green S.

Introduction

The relative safety of a colouring material in food may be judged by the satisfactory outcome of approved experimental investigation. Artificial food colourants are used in great amounts. Several Scientists and consumer

organizations have raised the question as to , whether artificial colourants should be allowed at all (Henry, 1980). Some investigators examined synthetic as well as natural colourants on certain biochemical items (Kiso et al., 1983 and Levitan et. al., 1984).

Chlorophyll is mainly present in the chloroplasts of higher plants. Two forms of chlorophylls (a , b) are usually found in a ratio of 3:1 . Some of synthetic dyes have been used as food colourants, but some of these possessed potential hazard to human health. Green S (wool Green BS, CI (1971) No. 44090; EECE. 142) is a water-soluble dye used widely by the food industry. It is the monosodium salt of 4, 4-bis (dimethylamino) - diphenylmethylen - 2 - Naphthol - 3 , 6 - disulphonic acid. Following the oral administration of the colourant at a dose level between 250 and 400 mg/kg to rats, showed that approximately 30% of the dye was recovered in the feces and up to 0.34% in the urine. No evidence for metabolism of the dye in these species (Phillips et. al., 1980).

The present study aimed at the investigation of the biological effects in male albino rats administered diet mixed with chlorophyll as a natural green pigment and Green S as a synthetic green colourant to evaluate their effects on carbohydrate, protein and lipids. Also liver function test was performed by determination of serum aspartate transaminase, alanine transaminase and lactate dehydrogenase activities. Moreover, histological studies were performed on liver samples for normals and rats which were fed by artificial dye as well as rats fed by natural pigments.

Material and Methods

Eighteen male albino rats, weighing 90 - 150 g, raised individually in a well aerated cages, under hygienic conditions for 60 days. The rats were fed balanced diet as described by El-Shobaky and Saleh (1985). They were divided into three groups (each of six rats). The first was kept as controls (fed colourant free diet) The second and third groups fed their diets supplemented with one gram of chlorophyll and of Green S pigment / kg diet, respectively. After 60 days and overnight fast, rats were sacrificed, blood samples were collected, sera were separated and livers were dissected out.

Total proteins and their electrophoretic separated fractions were determined as described by Ibrahim (1970). Serum glucose and liver glycogen were determined as described by Trinder (1969), and Carol et. al.,

(1955). Liver function test was investigated by estimating serum aspartate transaminase, alanine transaminase and lactate dehydrogenase activities using Biochemical Kits (Boehringer Mannheim GmbH, W. Germany). Total lipids, total cholesterol and phospholipids were determined as described by Knight et. al., (1972), Watson (1960) and Connerty et. al., (1961). Serum triacylglycerols were determined by the colorimetric method using Laboratory Kits from Wiener Lab. (2000 - Rosario - Argentina). Student's t - test was used to analyse the significance of experimental rat serum constituents compared to the mean control levels (Armitage, 1974). In all analyses a probability level of less than 5% was considered statistically significant. From each group of experimental rats, liver specimens were taken and fixed in formal saline 10% for histological study as described by Drury and Wallington (1967).

Results and Discussion

Green S consists of three linked aryl (usually phenyl) moieties with a central carbon atom. They all contain sulphonic acid groups which render them highly water-soluble and their low pKa values are held to be responsible for a characteristic low degree of absorption from gastrointestinal tract (Drake, 1975). No data are available on the metabolic fate of triphenylmethane dye in man. However, the principal factors governing absorption of dyes in both man and animals are the degree of ionization of dyes in the gut, lumen, the extent and nature of degradation effect by gut flora. Furthermore, the pH of the various regions of the normal human gut is substantially similar to that of the rat (Crampton, 1970) and it would be reasonable, therefore to expect that the biological fate of the dyes would be similar in man and the species investigated.

Phillips et. al., (1980) reported that, ¹⁴C-labelled Green S was not absorbed when orally given to rat or guinea-pig at a dose level of 30 µg/kg to 10 mg/kg and is not metabolised in the gastro-intestinal tract of these species. They found also that prolonged feeding of higher dose levels have no significant effect. Therefore, the lack of absorption and metabolism of Green S following ingestion in the rat, mouse and guinea-pig may account for the very low order of toxicity.

Body weight gain, liver weight and liver glycogen were markedly decreased while blood glucose was markedly increased as compared to

controls, on administration of either Chlorophyll or Green S (Table 1). There are slight variations between these items for rats fed chlorophyll and Green S ($P^{**} > 0.05$). Also, there were a slight decrease of total cholesterol, phospholipids and triacylglycerols. Total lipids were significantly decreased for the Green S group as compared to controls (Table 2). Green S is an effective hypolipidemic compound than chlorophyll ($P^{**} < 0.05$). El-Desoky (1993) reported that the natural pigments, Chlorophyll and Curcumen have powerful hypocholesterolemic and hypolipidemic effects. The hypocholesterolemic activity may be due to stimulation of lipolysis. He reported also stimulation of transaminases by pigment induction. These finding led to suggestion that cell metabolism was turned to glucose synthesis and decrease of glycogen. This is in accordance with our experimental results (Table 1).

Lactate dehydrogenase was significantly inhibited on administration the natural or synthetic pigments ($P^* < 0.05$). Feeding diets containing Chlorophyll or curcumen resulted in marked decrease of LDH activity (El-Desoky, 1993). AST and ALT activities in both plasma and liver homogenate were stimulated by Chlorophyll and Green S (Abdel Rahim et. al., 1987). However, ALT is increased in liver damage, While AST and occasionally ALT activities are increased in progressive muscular dystrophy. The significant inhibition of LDH on pigment feeding may be a result of unfavourable condition of liver, which cannot synthesize it. LDH is inhibited by reagents that react with thiol groups, Borate and oxalate inhibit it by competing with lactate for binding site on the enzyme. Excess pyruvate and lactate inhibit enzyme activity (Tietz, 1987). The lactate and pyruvate contents in different organs were dependent on the rate of glycolysis, pyruvate kinase and lactate dehydrogenase activities.

Clode et. al., (1987) reported low values of LDH activity, which do not indicate any biological damage. They considered that most of the differences from control encountered in their study do not represent any toxic effects on administration of Green S .There were a slight transitory anaemia , mild histological changes of the thyroid gland and slight nephro-histological changes. All of these were confined to the highest dose level and on this basis, they concluded that the Green S dose up to 500 mg/kg/day without toxic effects.

Administration of Chlorophyll or Green S resulted in significant increase of serum total proteins and gamma-globulin, but resulted in slight variations of albumin, alpha1-, alpha2- and beta-globulins as compared to controls (Table 4).

Feeding with Green S has variable slight effect on serum proteins fractions as compared to the effect of Chlorophyll feeding. The increase of alpha-globulins is a noteworthy feature of acute febrile diseases, which resulted in increased tissue mucopolysaccharides breakdown or their excessive formation. The marked hypergammaglobulinemia is due to antibodies formation and / or due to regeneration of reticuloendothelial system to synthesize globulins.

Abdel-Rahim et. al., (1987) reported that colourants might regulate plasma proteins and liver soluble fraction by stimulating protein biosynthesis to produce specific enzymes to protect these conditions (for detoxication) of natural and synthetic pigments. This increase is still in the normal range. But this cannot be as a permission for continuous consumption of such compounds especially the synthetic one. Ibrahim et. al., (1988) reported an increase of T3 and T4 hormones excretions. The elevation of serum proteins as a result of Green S and Chlorophyll induction may be due to increase of DNA and RNA that stimulate protein biosynthesis.

Histological studies showed that parenchyma of control liver and liver of rats fed diet supplemented with Chlorophyll consisted of radiating cords of hepatocytes. The hepatocytes appeared polyhedral in shape with acidophilic cytoplasm. The nuclei were rounded and centrally located. The blood sinusoids appeared as narrow spaces between the hepatic cords (Fig.1). Williams (1959) reported that the information ideally needed about food colourants, includes knowledge of its toxicity, absorption, metabolism, excretion and accumulation. Our histological results were mainly found with rats receiving Green S . The inflammatory foci seen in the hepatic parenchyma showed a higher incidence in rats fed with synthetic dye . These might be due to the toxic effect of this dye on hepatocytes (Fig. 2, 3, 4). This is in accordance with results of Moorhouse et. al., (1987), but they did not consider these changes to be of any toxicological significance.

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Table 1: Effect of chlorophyll and Green S Administration on body weight Gain, Liver Weight, Blood Glucose and Liver Glycogen

	body weight gain (g)	liver weight (g)	blood glucose (mg/dl)	liver glycogen (g%)
a- Rats fed diet free colourants (controls).				
mean \pm SE.	192.5 \pm 11.05	9.33 \pm 0.66	94.77 \pm 0.89	5.09 \pm 0.16
b- Rats fed diet + Chlorophyll (1g / kg diet).				
mean \pm SE.	149.8 \pm 12.14	7.66 \pm 0.58	115.64 \pm 3.06	3.21 \pm 0.27
P* <	0.05	0.05	0.05	0.05
c- Rats fed diet + Green S (1g / kg diet).				
mean \pm SE.	163.6 \pm 9.09	7.22 \pm 0.84	116.29 \pm 4.23	3.00 \pm 0.05
P* <	0.05	0.05	0.05	0.05
P** <	N.S.	N.S.	N.S.	N.S.

P* : P values less than 0.05 is considered significant on comparison to controls.

P** : P values less than 0.05 is considered significant on comparison between synthetic and natural colourant

N.S. : not significant

Table 2 : Effect of chlorophyll and Green S Administration on total lipids, total cholesterol, Phospholipids and Triacylglycerols (mg / dl).

	total lipids	total cholesterol	phospholipids	triacylglycerols
<u>a- Rats fed diet free colourants (controls).</u>				
mean \pm SE.	832.9 + 25.64	105.7 + 8.13	132.0 + 10.8	51.0 + 5.1
<u>b- Rats fed diet + Chlorophyll (1g / kg diet).</u>				
mean \pm SE.	764.8 + 39.9	104.0 + 0.41	115.3 + 10.2	44.8 + 2.57
P* <	N.S.	N.S.	N.S.	N.S.
<u>c- Rats fed diet + Green S (1g / kg diet).</u>				
mean \pm SE.	657.0 + 26.21	92.0 + 5.46	114.1 + 10.0	44.0 + 2.02
P* <	0.05	N.S.	N.S.	N.S.
P** <	0.05	N.S.	N.S.	N.S.

P* : P values less than 0.05 is considered significant on comparison to controls.

P** : P values less than 0.05 is considered significant on comparison between synthetic and natural colourant

N.S. : not significant

Table 3 : Effect of chlorophyll and Green S Administration on Serum AST , ALT and Lactate dehydrogenase Activities (U / l)

	AST	ALT	LDH
<u>a- Rats fed diet free colourants (controls).</u>			
mean \pm SE.	43.42 \pm 0.87	16.13 \pm 1.92	383.3 \pm 3.58
<u>b- Rats fed diet + Chlorophyll (1g / kg diet).</u>			
mean \pm SE.	45.3 \pm 2.42	17.92 \pm 2.30	274.2 \pm 3.01
P ^a <	N.S.	N.S.	0.05
<u>c- Rats fed diet + Green S (1g / kg diet).</u>			
mean \pm SE.	45.45 \pm 1.81	19.8 \pm 3.36	269.6 \pm 10.8
P ^a <	N.S.	N.S.	0.05
P ^{a*} <	N.S.	N.S.	N.S.

P^a : P values less than 0.05 is considered significant on comparison to controls.

P^{a*} : P values less than 0.05 is considered significant on comparison between synthetic and natural colourant

N.S. : not significant

Table 4 : Effect of chlorophyll and Green S Administration on Serum Protein Fractions (g / dl)

	T. P.	Alb.	α_1 -g	α_2 -g	β -g	δ -g
<u>a- Rats fed diet free colourants (controls).</u>						
mean \pm SE	7.22 \pm 0.16	4.54 \pm 0.09	0.58 \pm 0.05	0.57 \pm 0.03	0.55 \pm 0.05	0.98 \pm 0.07
<u>b- Rats fed diet + Chlorophyll (1g / kg diet).</u>						
mean \pm SE	7.73 \pm 0.23	4.56 \pm 0.14	0.64 \pm 0.09	0.42 \pm 0.08	0.67 \pm 0.06	1.28 \pm 0.10
P ^a <	0.05	N.S.	N.S.	N.S.	N.S.	0.05
<u>c- Rats fed diet + Green S (1g / kg diet).</u>						
mean \pm SE	7.82 \pm 0.11	4.78 \pm 0.14	0.42 \pm 0.11	0.74 \pm 0.05	0.45 \pm 0.13	1.29 \pm 0.10
P ^a <	0.05	N.S.	N.S.	N.S.	N.S.	0.05
P ^{bc} <	N.S.	N.S.	N.S.	0.05	N.S.	N.S.

T.P : total proteins , Alb : albumin , α_1 -g : alpha1 -globulin , α_2 -g : alpha2-globulin , β -g : beta - globulin , δ -g : gamma -globulin

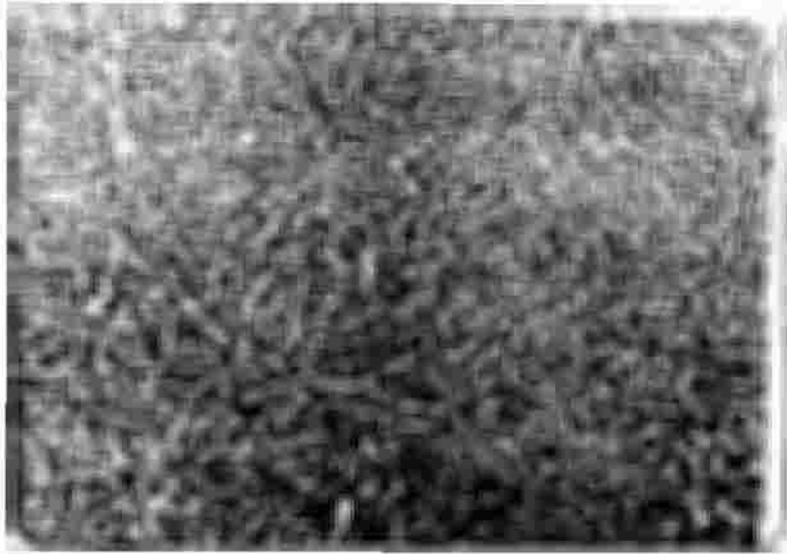


Fig. 1: Normal control liver. H & E stain, Mic. Mag. X 100

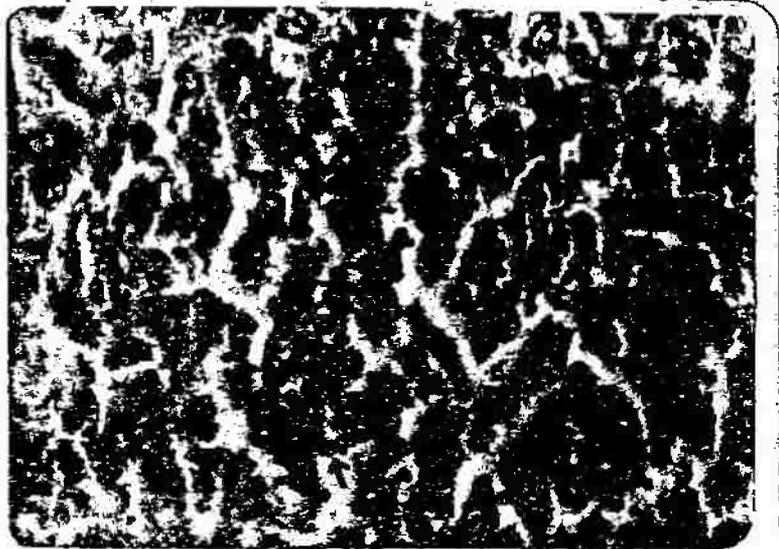


Fig. 2: Liver of rats fed Green S, showing degenerated hepatocytes in some areas. Streaks of mononuclear cellular infiltration were evident (arrow). Mic. Mag. X 400

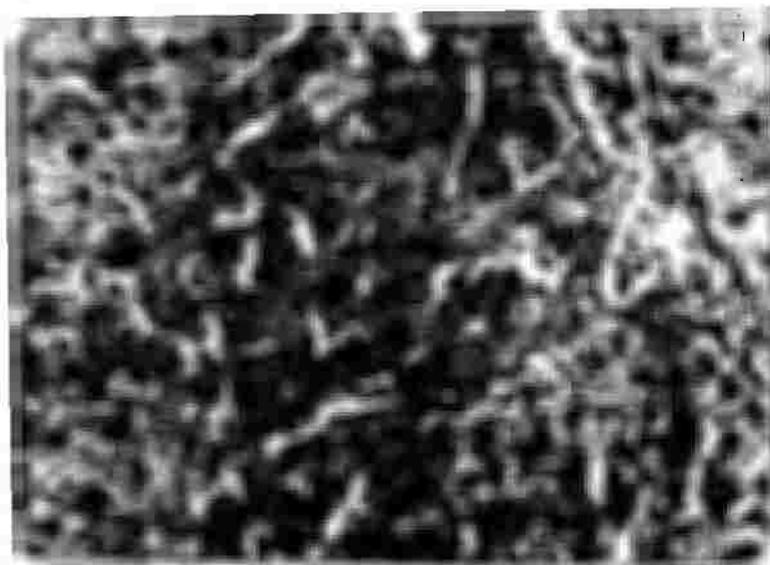


Fig. 3: Liver of rats fed Green S, showing focal degenerated hepatocytes were apparent. Moderate cellular reaction was evident (arrow). Mic. Mag. X 400

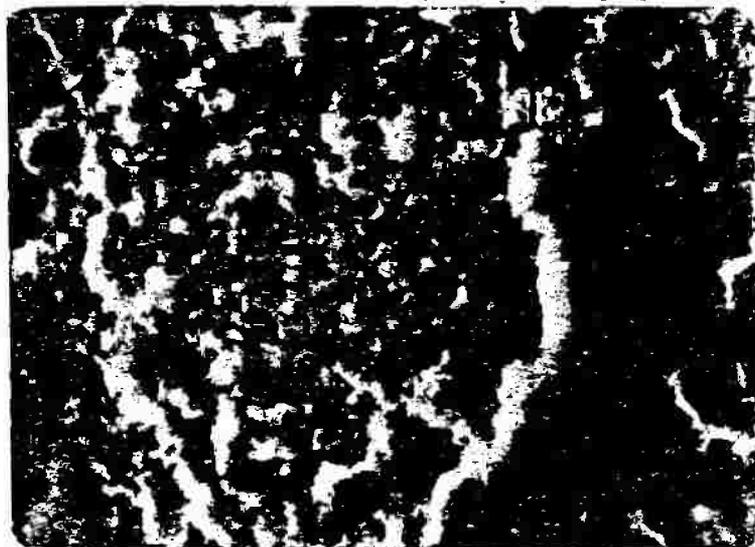


Fig. 4: Liver of rats fed Green S, showing focal aggregations of cellular reactions pressing the surrounding hepatocytes causing some degenerative changes (arrow). Mic. Mag. X 400

مقارنة بيولوجية عن تأثير التغذية بالكوروفيل و الـ Green S في الفئران البيضاء

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تم دراسة تغذية مادتي الكوروفيل و الـ Green S على التمثيل الغذائي للكربوهيدرات و البروتينات و الليبيدات. أظهرت أوزان الفئران و أكبادها و كذلك مستوى جليكوجين الكبد إنخفاضاً واضحاً بينما يرتفع مستوى جلوكوز الدم. إنخفضت مستويات الدهون الكلية و الكوليسترول و الفسفوليبيدات و ثلاثي أسيل جلسيرول إنخفاضاً طفيفاً. أدت التغذية بمادة Green S إلى إنخفاض مستوى الدهون الكلية بوضوح بمقارنتهم بالفئران الضابطة و التي تغذت على الكوروفيل. إزدادت أنشطة الإنزيمات الناقلة لمجموعة الأمين كما أدت للتغذية بالمونيات إلى إعلاء نشاط إنزيم فلاككات ديبيدروجينيز. زاد مستوى البروتينات الكلية و الجاما جلوبيولينات بوضوح بينما أظهرت مستويات الألبومين، الألكا، ألبينا جلوبيولينات تغيرات طفيفة مختلفة.

صاحبت التغيرات الهرمستروية في كبد الفئران التي تغذت على ملون Green S .
أظهرت بؤر ملتهبة في أنسجة كبد الفئران التي تغذت على الصبغة الصناعية و الذي يرجع لتأثيرها السام.

Synthesis and Some Reactions of New Quinazolone Derivatives

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2-(2-p-AnisoylvinyI)-4H-3,1-benzoxazin-4-one (I) easily condensed with *o*-toluidine and/or *p*-phenylene-diamine affording the formation of the corresponding quinazolone derivatives (IIa and b).

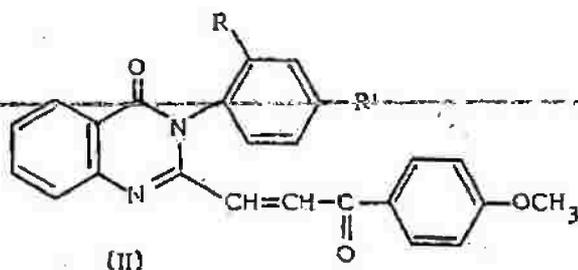
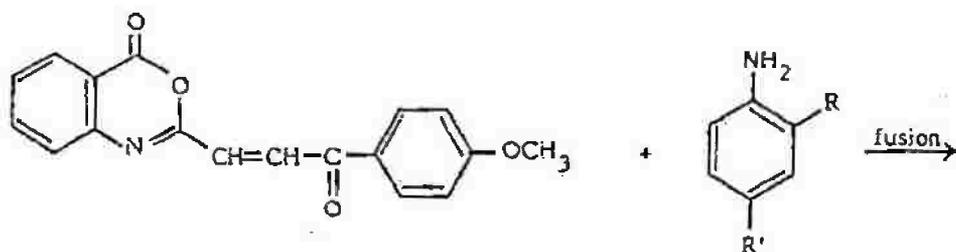
Reaction of quinazolones IIa and b with Br₂, amines, aromatic aldehydes, hydrazines, urea and thiourea has been investigated.

2-(2-AroylvinyI)-4H-3,1-benzoxazin-4-one has been prepared^(1,2) by the interaction between ethereal solution of anthranilic acid with acryloyl chloride followed by cyclization of the product via boiling with acetic anhydride.

In the present investigation, we would like to report on the synthesis of a number of new 2,3-disubstituted-4-quinazolones for the object of their pharmacological evaluation^(3,4).

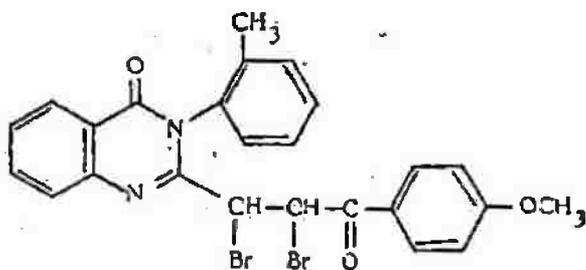
Thus condensation of 2-(2-p-anisoylvinyI)-4H-3,1-benzoxazin-4-one (I) with *p*-toluidine and/or *p*-phenylene diamine^(5,6) afforded the formation of 2-(2-p-anisoylvinyI)-3-*o*-tolyl)-4(3H)-quinazolinone (IIa) and 2-(2-p-anisoylvinyI)-3-(4-aminophenyI)-4(3H)-quinazolinone (IIb) respectively.

(2)



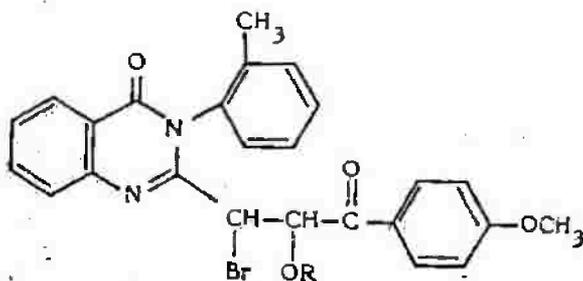
R	R'
a, CH ₃	H
b, H	NH ₂

The bromination of IIa in chloroform afforded the corresponding dibromide derivative III.



(3)

Refluxing of the dibromide III with alcohols, namely, methyl and/or ethyl alcohol afforded the formation of the corresponding α -bromo- β -methoxy or ethoxy derivatives IVa and b respectively. (7)

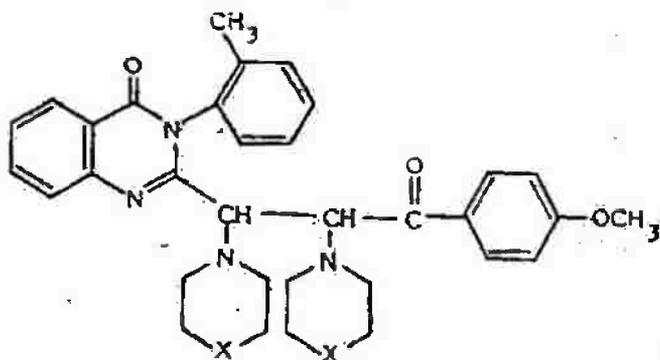


(IV)

a, R = CH₃

b, R = C₂H₅

On the hand the reaction morpholine and/or piperidine reacts with the dibromide III gave the corresponding α , β -dimorpholino and α - β -dipiperidino derivatives Va and b, respectively.



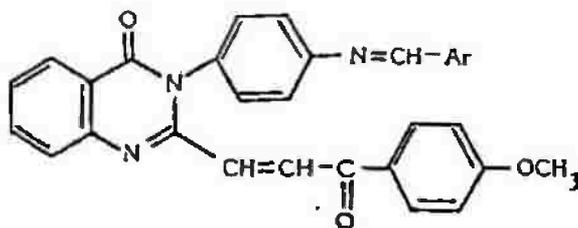
(V)

a, X = O

b, X = CH₂

(4)

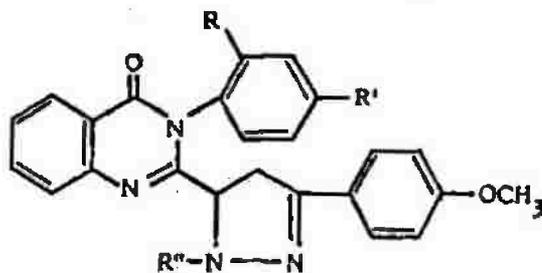
Condensation of 2-(2-p-anisoylvinyl)-3-(4-aminophenyl)-4 (3H)-quinazolinone (IIb) with aromatic aldehydes namely benzaldehyde, anisaldehyde and/or p-chlorobenzaldehyde afforded the formation of the corresponding azomethines derivatives VIa-c respectively.



(VI)

Ar = C₆H₅ (a); Ar = C₆H₄OCH₃ (p) (b) and Ar = C₆H₄-Cl (p) (c)

As a point of interest IIa and b condensed easily with hydrazine hydrate and/or phenylhydrazine in boiling ethanol affording the formation of the pyrazolonyl quinazolinone derivatives VIIa-d, respectively.



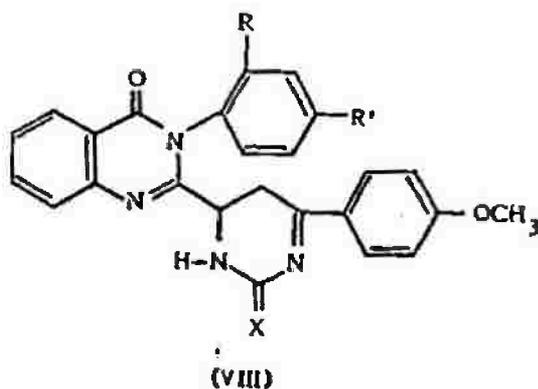
(VII)

	R	R'	R''
a	CH ₃	H	H

(5)

b	CH ₃	H	C ₆ H ₅
c	H	NH ₂	H
d	H	NH ₂	C ₆ H ₅

The synthesis of oxo- and thioxo-pyrimidino derivatives VIIIa-d respectively was investigated.



	R	R'	X
a,	CH ₃	H	O
b,	CH ₃	H	S
c,	H	NH ₂	O
d,	H	NH ₂	S

EXPERIMENTAL

The infrared absorption spectra were determined with a Unicam SP 1200 Spectrophotometer using KBr wafer technique and listed with other physical data in Table 1.

The NMR spectra were obtained using Varian Model A-90 spectrophotometer and listed in Table 2.

All melting points are not corrected.

Reaction of 2-(2-p-anisoylviny)-4H-3,1-benzoxazin-4-one (I) with o-toluidine.

Formation of IIa:-

A mixture of 2-(2-p-anisoylviny)-4H-3,1-benzoxazin-4-one (I) (0.01 mole), o-toluidine (0.01 mole) and anhydrous $ZnCl_2$ (0.5 g) was heated in an oil bath at 150-160°C for 4 hrs. The solid product was cooled, washed with dilute HCl (5%) filtered off and crystallized from ethanol to give the corresponding quinoxalinone derivative IIa as yellow crystals melted at 187-8°C. Yield 75%.

Reaction of (I) with p-phenylene diamine. Formation of IIb.

A mixture of 2-(2-p-anisoylviny)-4H-3,1-benzoxazin-4-one (I) (0.01 mole) and p-phenylene diamine (0.01 mole, 1.08 g) was heated on a direct flame for 10 minutes. The reaction product was cooled and triturated with ethanol and filtered off. The crude solid product thus obtained was recrystallized from ethanol to give IIb as yellow crystals melted at 205°C. Yield 60 %.

(7)

Bromination of IIa. Formation of the dibromide III:-

To a cold solution of IIa (0.01 mole) in chloroform was added gradually a chloroform solution of bromine (0.01 mole) and the reaction mixture was stirred for 1 hr. The solvent was evaporated and the separated solid was collected and re-crystallized from benzene / ethanol mixture (5:1) to give the corresponding dibromide derivative III as colourless crystals melted

at 248°C. Yield-85%.

Reaction of III with alcohols. Formation of IVa and b:-

A mixture of III (1 g) and methyl or ethyl alcohol (50 ml) was refluxed for 1 hr. The solution concentrated to half its volume, cooled and the obtained solid products were crystallized from the appropriate solvent to give the corresponding α -bromo- β -methoxy or ethoxy derivatives IVa and b, respectively.

Reaction of III with morpholine and piperidine. Formation of Va and b:-

A mixture of III (1 g), morpholine and / or piperidine (2 ml) and (25 ml) benzene was heated under reflux for 2 hrs., filtered while hot and concentrated. The solid product obtained was crystallized from the appropriate solvent to give the corresponding dimorpholino or dipiperidino derivatives Va and b, respectively.

Condensation of IIb with aromatic aldehydes. Formation of VIa-c:-

A mixture of IIb (0.01 mole), benzaldehyde, anisaldehyde and/or *p*-chlorobenzaldehyde (0.01 mole), ethanol (30 ml) and glacial acetic acid (0.5 ml) was refluxed for 4 hrs. The solid

(8)

product obtained after concentration and cooling was crystallized from proper solvent to give VIa-c, respectively.

Condensation of IIa and b with hydrazines. Formation of VIIa-d:-

A mixture of IIa and/or b (0.01 mole), hydrazine hydrate and/or phenylhydrazine (0.01 mole) and ethanol (20 ml)⁶ was refluxed for 4 hrs. The solid product obtained after removal of the most of the solvent was crystallized from proper solvent to give the pyrazoline derivatives VIIa-d, respectively.

Reaction of IIa and b with urea and/or thiourea. Formation of VIIIa-d :-

A mixture of IIa and/or b (0.01 mole), urea and/or thiourea (0.01 mole) and ethanol (20 ml) was treated with 10 drops of glacial acetic and refluxed for 6 hrs. The solid product formed after removal of the of the solvent and cooling was crystallized from proper solvent to give the corresponding pyrimidinones and pyrimidine thiones VIIIa-d, respectively.

(9)

The physical properties and infrared of the new compounds

Compound	M.p.°C (Colour)	Solvent (Yield)	Formula (M.Wt.)	Analysis %		Group frequencies (KBr, cm ⁻¹)
				Found	Require	
IIa	187-8 (yellow)	E 75 %	C ₂₅ H ₂₀ O ₃ N ₂ (396)	C 75.32 H 4.91 N 6.88	75.75 5.05 7.07	C=O 1700, 1680 C=O 1580 C=N 1630
IIb	205 (yellow)	E E 60%	C ₂₄ H ₁₉ O ₃ N ₃ (407)	C 70.15 H 4.25 N 12.40	70.76 4.66 12.77	C=O 1720, 1675 C=C 1580 C=N 1620 NH 3380(broad)
III	248 colourless	B/E 85%	C ₂₅ H ₂₀ O ₃ N ₂ Br ₂ (576)	C 51.46 H 2.28 N 3.97 Br 30.84	52.08 3.47 4.68 31.25	C=O 1710, 1630 C=C 1590 C=N 1630
IVa	210-1 colourless	E 50%	C ₂₆ H ₂₃ O ₄ N ₂ Br (517)	C 59.77 H 4.40 N 5.11 Br 16.85	60.34 4.44 5.41 17.40	C=O 1710, 1690 C=C 1625
IVb	227-8 colourless	E 70%	C ₂₇ H ₂₅ O ₄ N ₂ Br (531)	C 60.32 H 4.05 N 4.76 Br 16.32	61.01 4.70 5.27 16.94	C=O 1735, 1680 C=N 1630
Va	180-1 colourless	B 65%	C ₃₃ H ₃₆ O ₅ N ₄ (568)	C 69.54 H 5.57 N 9.27	69.97 6.33 9.85	C=O 1730, 1695 C=N 1630
Vb	200-1 colourless	E 70%	C ₃₅ H ₄₀ O ₃ N ₄ (564)	C 73.78 H 6.66 N 9.27	74.64 7.09 9.92	C=O 1705, 1675 C=N 1625

Table 1 (Cont. . .)

V Ia	154-6 (yellow)	E 65%	$C_{31}H_{23}O_3N_3$ (485)	C 75.84 H 4.56 N 8.42	76.70 4.74 8.65	C=O 1725, 1680 C=N 1630, 1645
V Ib	177-8 (yellow)	B 50%	$C_{32}H_{25}O_4N_3$ (515)	C 73.81 H 4.22 N 7.62	74.56 4.85 8.15	C=O 1710, 1665 C=N 1635, 1620
V Ic	203-4 (yellow)	E 55%	$C_{31}H_{22}O_3N_3Cl$ (519.5)	C 71.21 H 3.93 N 7.86 Cl 6.51	71.60 4.23 8.08 6.83	C=O 1745, 1705 C=N 1640, 1620
V IIa	184-5 (yellow)	A 60%	$C_{25}H_{19}O_2N_4$ (407)	C 73.18 H 4.06 N 13.47	73.71 4.66 13.75	C=O 1680 C=N 1635 NH 3380 (Broad)
V IIb	196-7 (yellow)	B/E 50%	$C_{31}H_{23}O_2N_4$ (483)	C 76.84 H 3.52 N 10.76	77.01 4.76 11.59	C=O 1680 C=N 1630, 1650
V IIc	132-3 (yellow)	B 55%	$C_{24}H_{21}O_2N_5$ (411)	C 69.18 H 4.74 N 16.60	70.07 5.10 17.03	C=O 1665 C=N 1640 NH 4080
V II d	167-8 (yellow)	E 70%	$C_{30}H_{25}O_2N_5$ (487)	C 73.22 H 4.76 N 13.87	73.92 5.13 14.37	C=O 1660 C=N 1640 C=N 3985
V IIIa	159-60 (yellow)	E 60%	$C_{26}H_{19}O_3N_4$ (435)	C 71.02 H 3.97 N 12.24	71.72 4.36 12.87	C=O 1685 C=N 1640 NH 3890

Table I (cont. . .)

VIIIb	227-8 (yellow)	A 40%	$C_{26}H_{19}O_2N_4S$ (451)	C 68.77	69.17	C=O 1670
				H 3.84	4.21	C=N 1640
				N 12.14	12.41	C=S 1340
				S 6.82	7.09	NH 4225
VIIIc	144-5 (yellow)	B 65%	$C_{25}H_{21}O_3N_5$ (439)	C 68.06	68.33	C=O 1680
				H 4.42	4.78	C=N 1635
				N 15.62	15.94	NH 4180
VIIId	261-2	E 40%	$C_{25}H_{21}O_2N_5S$ (455)	C 65.44	65.93	C=O 1660
				H 4.18	4.61	C=N 1630
				N 15.15	15.38	C=S 1360
				S 6.85	7.03	NH 4040

E = Ethanol

B = Benzene

Table (2)

The N.M.R. spectra of some new compounds

Compound	Solvent	δ -values	Group
IIa	CDCl ₃	6.65-7.85	12 aromatic hydrogen
		6.50	1 H of $-\text{CH}=\text{CH}-\overset{\text{O}}{\parallel}{\text{C}}-$
		6.40	1 H of $-\text{CH}=\text{CH}-\overset{\parallel}{\text{C}}-$
		4.40	3 H of $-\text{OCH}_3$
		2.25	3 H of $-\text{CH}_3$ in phenyl group
IIb	(CD ₃) ₂ CO	6.95-7.40	12 aromatic hydrogen.
		6.65	1 H of $-\text{CH}=\text{CH}-\overset{\text{O}}{\parallel}{\text{C}}-$
		6.45	1 H of $-\text{CH}=\text{CH}-\overset{\parallel}{\text{C}}-$
		4.25	3 H of $-\text{OCH}_3$
		8.16	2 H of $-\text{NH}_2$
VIIIa	DMSO	6.90-7.95	18 aromatic hydrogen
		2.50	2 H $-\text{CH}_2$ in pyrazoline
		6.65	1 H of $-\text{CH}$ in pyrazoline
		2.30	3 H of $-\text{CH}_3$ in phenyl group
		4.20	3 H of $-\text{OCH}_3$ group.
VIIIa	CDCl ₃	6.85-7.55	12 aromatic hydrogen
		6.90	1 H of $-\text{CH}$ in pyrimidinone
		2.60	2 H of $-\text{CH}_2$ in pyrimidinone
		3.65	1 H of $-\text{NH}$ in pyrimidinone
		2.45	3 H of $-\text{CH}_3$ group
		4.40	3 H of $-\text{OCH}_3$ group

ACTIVITY OF QUINAZOLONES

Quinazolones IVa,b exhibit pronounced CNS depressant, anticonvulsant and tranquilizing effects.

While, quinazolones VIa,b possess antifertility activity, muscle relaxant, antihelminthic activity against M-dubius infection in mice.

Quinazolones VII successfully screened as antihypertensives, antibrillatory, antiphlogistic, hypotensive, antiadrenergic, herbicidal and bactericidal agents.

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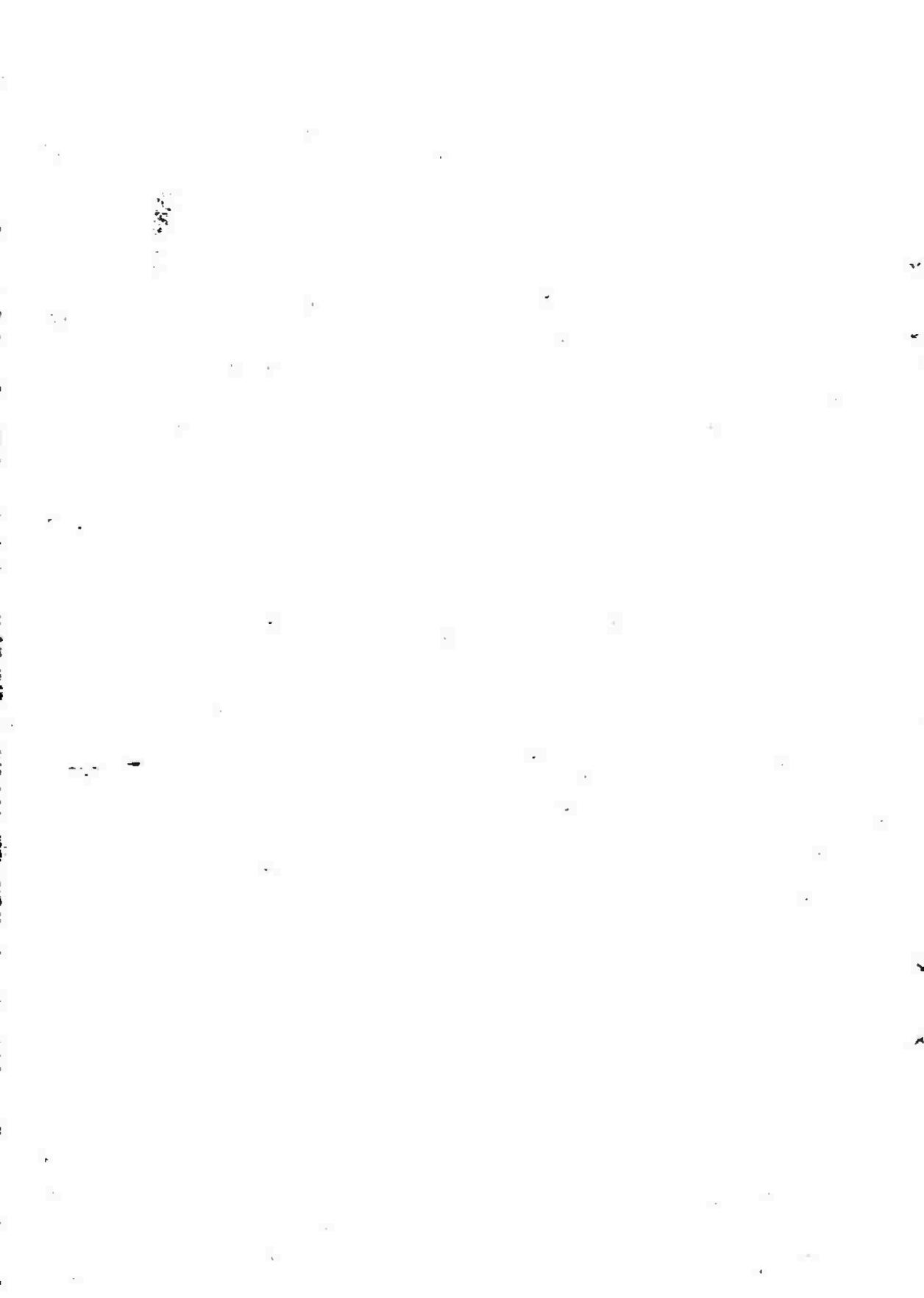
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تحضير بعض التفاعلات مشتقات الكينازولون الجديدة
طه محمود عبد الرحمن - كلية التربية النوعية - العباسية
القاهرة

لخص:

يتكافئ ٢ - (٢ - بارا - أنيزيل فينيل) - ٤ يد - ١٣ - نيزوالزين ٤ - ون
(I) مع بارا طولويدين وبارا فينيلين ثنائي الاين معطيا مشتقات الكينازولون
(II a, b) .

وتقد تم استحداث تفاعل الكينازولونات (II a, b) مع البروم ، الاينسات ،
الدهيدات الأروماتية والهدرازينات واليوريا والتيوريا .



CONTENTS

	<u>Page</u>
1 - Synthesis of Triazolo (4,3-b) Pyridazines. By Eliazi I. Al-Afaleg.....	1
2 - Inhibiting Effect of triphenyl tetrazolium Chloride(TTC) on the Acid corrosion of cadmium. By Ensherah Abdel - Wahab Abdel - Hamed.....	6
3 - Inhibition of Acid corrosion of cadmium by Sodium molybdate. By Amira A. Misbah.....	21
4 - Protection of Nickel Metal by Vinyl Polymer in Nitric Acid. By Omya R. Khalifa.....	35
5 - Some Reactions with 6-Bromo-2-phenyl-3,1-benzoxazin-4- one. By S.A. Emar.....	48
6 - Some Reactions With 6-Bromo-2-phenyl-3,1-benzoxazin-4- one. By S.A Emar.....	60
7 - Synthesis and Some Reactions of 6-(4-Methoxyphenyl)-4- (3,4-dimethoxyphenyl-methyl)-pyridazin-3(2H)-one. By A.F. Elkafrawy.....	72
8 - Studies on some transition metal chloranilates. By Marguerite A. Wassef, Nagwa H. Esmail and Azza A.Abu Hessein,.....	83
9 - Synthesis and Some Reactions of 4-(3,4-Dimethoxybenzyl) -6-phenylpyridazin-3(2H)-one. By Omnia E.A. Mustafa.....	103
10- Spectroscopic studies of Electron Donor-Acceptor Chromogens of Oxazolin-5-one Derivatives. By Yvette, A. Issac.....	115
11- A Novel poly (Vinyl chloride) Matrix membrane Electrode for the selective determination of buformin. By Mona A. Ahmed.....	127
12- Fluorescence quenching of fluorene and azafluorene compounds by some pyridine and quinoline N-Oxide Derivatives. By M.M. Abo Aly.....	140

	<u>page</u>
13- Anodic Dissolution of lead in Acid Solution. By A.A. Misbah, Z.A. El Hady and O.R. Khalifa.....	152
14- Corrosion Behaviour of Lead in Solutions of Mixed Anions. By A.A. Misbah, Z.A. El Hady and O.R. Khalifa.....	166
15- ¹ H NMR (400 MHz) Spectroscopic Studies of some (Z) and (E) Esters and their Isomeric Derivatives . By N.R. Guirguis , B.M. Awad and S.M. Abdallah.....	187
16- Evaluation of Local Clay as Dehydration Catalyst. By Suzan A. Ali , Maha K. El Aiashy and Zita S. Ayad.....	199
17- Reaction of Thiosemicarbazide with Acetylenic Ketones and Esters . By Ahmed Said Ahmed Youssef.....	217
18- Dimer Fatty Acid-Modified polyesteramide Resin Compositions. By A. A. Borayi	227
19- Statistical Analysis of VHF Signal Transmission in Egypt and Across Red Sea. By A. El Bialy, E. Aziz, A. M. Swidan and S.M. El Ghanaw..	240
20- Solar Activity -Sunspot Number- Effect on High Frequency Electromagnetic Wave Propagation. By A. M. Swidan.....	252
21- Stability of Aflatoxin M ₁ During Manufacture and Storage of Yoghurt-Cheese and Acidified Milk. By Nagwa I. Hassanin.....	265
22- Role of Trace Elements in Aflatoxin Production by <i>Aspergillus Flavus</i> . By Adel Ahmed El Mehalawy.....	278
23- The effect of some growth conditions on the production of lipases by <u>Azotobacter Chroococcum</u> , <u>Rhizobium</u> <u>sesbani</u> and <u>R. lupini</u> , 282. By M.S.Ammar , M.S. El Gamal, S.S. El Louboudy and M.K. El Mousallamy	284
24- Nutritional Requirements and Invertase Activity of <u>Rhizobium</u> Nodulating <u>Sesbania Sesban</u> Roots. By Fatma A. Helemish, Mehreshan T. El Mokadem and Samia H. Abou Zekry	301

25-	The interactive effects of CaSO_4 and NaCl on the Growth and nodulation of <u>Pisum Sativum</u> . By Fatama A. Helemish	312
26-	The effects of Copper levels and Nitrogen sources on nodultion, dry matter yield, Copper and Nitrogen contents of <u>Vicia faba</u> under greenhouse condition . By Fatma A. Helemish	328
27-	Effect of some seed Hardening Treatments on Chemical composition of sudan grass grown under saline conditions. By Safaa M. Ismaeil, Om Mohamed A. Khafaji, E. T. Kishk and Samya M. Sohsah	338
28-	Cytogenetic Response of <u>Vicia faba</u> plants to the Herbicide " Treflan" . By Enaam M. Ali	358
29-	Competition as a biotic ecological factor. By P.G.M.A.Khadre	374
30-	Chromosomal Changes in <u>Allium cepa</u> induced by <u>Ruta graveolens</u> water extract. By A.I. El Mahas , A.S. Shehab and H. Megazi	396
31-	A study on salt sensitivity of two wheat durum cultivars: sohag and beni swaf. By Soad A. G. Sbetawi, M.A. Hammouda, Kawzar M.Tawfik and Zeinab Y.M. Abou Bakr	408
32-	Existance and growth of halophilic bacteria from hypersaline environments. By M.E.A.A. Abdel - Monem	423
33-	Toxicity of the pyrethroid insecticide decamethrin and fenvalerate to fresh - water clarias lazera. By Moreed Yanni , Hussein Badawi and Rokaya Saeed.....	439
34-	Toxicity of the pyrethroid insecticide decametrin and fenvalerate to a fresh water fish clarias lazera. By Moreed Yanni , Hussein Badawi and Rokaya Saeed.....	457
35-	Studies on low calorie ice milk 4-preparation of low calorie frozen yoghurt. By Zakia M. Abdel - Kader, Aziz E. Khader and Hamdia A.E. Halal	473