Experimental Modelling wax preparation from Petroleum products and natural additives

Running Title: Preparation Experimental Modeling Wax

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ABSTRACT

Introduction: Many modelling waxes are composed of complicated mixture of many constituents.

Aims of the study: To modify Petroleum Iraqi natural waxes (hard and soft paraffin wax, natural and commercial beeswax) by adding some natural (gum Arabic, rosin, Na-CMC, starch, and amaranth) and chemical (ferric oxide) to prepare new experimental modelling waxes that can be used in prosthetic dentistry. Evaluate physical properties according to ADA specification No. 24 and ISO 1561 in comparison to two commercials modelling waxes (Major® and Polywax™).

Materials and Methods: Total samples prepared are 478 samples, and divided into 3 groups: (1) Iraqi Petroleum natural waxes and commercial beeswax. (2) Iraqi Petroleum natural waxes mixtures, the mixtures are either binary (Hard + soft Paraffin) or tertiary mixtures (Beeswax). The binary mixtures are (90% + 10%) and (80%+20%).The Tertiary mixtures are (80% + 15% + 5%) and (70% + 20% + 10%). (3) Natural waxes and additives, the percentages of mixing are (90% natural waxes + 10 % additives) and (80% natural waxes + 20% additives). (100) samples have been failed in flow and thermal expansion test until obtained the proper percentage of mixing that give proper physical tests: melting point, flow, thermal expansion, and accuracy test. **Results**: there were significant differences ($p \leq 0.001$) between control and experimental modelling waxes {10 (80% hard paraffin + 20% beeswax) & (90% beeswax + 10% starch)}. The results showed that the addition of 20% Na-CMC, gum Arabic, Starch, and rosin to hard paraffin led to decrease the maximum melting point, flow, thermal expansion and dimensional changes but increase the minimum melting point**. Conclusions:** It was concluded that the experimental modelling waxes: (80% hard paraffin + 20% beeswax) and (90% beeswax + 10% starch) have the most nearest properties to control and ADA specification No. 24 than other waxes.

Key Words: Paraffin, Rosin, Starch, Melting point.

Introduction: Waxes form a group of thermoplastic materials which are normally solids at room temperature but melt, without decomposition, to form mobile liquids. They are, essentially, soft substances with poor mechanical properties and their primary use in dentistry is to form patterns of appliances prior to casting [(1)].

From the early use of beeswax in the eighteenth century for making impression to current techniques, waxes have always been among the most popular and useful of dental materials [(2,3)].

The utility of dental waxes stems from several factors: they are cheap, non-toxic, and low melting, weak solids that can be readily shaped and molded. They are used for some of the highest precision work in dentistry, as well as cruder tasks, yet they have the worst thermal expansion coefficient of all material used in dentistry [(4,5,6)]. Dental waxes are very complex mixtures, perhaps containing hundreds of compounds, mostly natural products. They are formulated by blending natural and synthetic waxes, plus minor components such as resins, oils, and pigments, to control the properties according to the intended use [(7,8)]. The ultimate goal of the combination of waxes and additives is to produce dental waxes that posses a set of given physical properties over specified range of temperature [(4).]

McCrorie[(9)] found that many modelling waxes are composed of complicated mixture of many constituents. It is also certain that the major component is paraffin wax with the addition of varying amounts of higher melting point waxes such as beeswax, carnauba wax and microcrystalline waxes.

McCrorie[(10)] reported that the modelling and baseplate waxes are the most widely used dental materials. The easiness of manipulation, good sculpting properties and simplicity of disposal of wax pattern, by boiling-out, probably account for much of the popularity of modelling wax as a pattern material[(9)].

Modelling waxes are used as a pattern material, for registration of jaw relationship and in construction of dentures [(2)]. Heath *et al.*[(11)] stated that the production of successful dentures necessitates the use of accurately fitting base plate

materials for recording of the jaw relationship and trial insertion of the waxed-up dentures.

Previously, two types of wax were formulated in Iraq, boxing wax [12] , and inlay dental wax[13] from the available wax purchased from Dura Refinery Center and other Iraqi sources.

MARTERIALS AND METHODS:

All the additives materials were prepared from the natural form as a powder (starch, Sodium carboxymethylcellulose (Na-CMC), Gum Arabic, and Rosin). The sieving procedure was done according to Abdullah [14] using a sieve 63 µm starts by weighing the material. The particle size of material is made homogenous (Figure 1). According to ADA[15], and ADA Specification No. 24 and ISO 1561[16] the coloring agent Amaranth have been added to wax mixtures in proportion of 0.02 % at 40°C incrementally and with continuous mixing until obtaining a uniform and desired color that coincides with the color of commercial modeling waxes checked by naked eye. The total number of samples that has been prepared is (478) samples. One hundred samples failed during flow and thermal expansion tests until determining the proper percentage of wax mixtures that give results coincide with control and ADA specification No. 24. The final number of samples that has been used is (378) samples (Table I).

The melting point was measured according to method reported by Vogel [17] using Electrothermal melting point apparatus (CE, VWR, INTERNATIONAL). One end of each of the capillary tubes had been sealed by inserting it horizontally into extreme edge of a small Bunsen flame for a few seconds and the capillary tube is rotated meanwhile. The prepared capillary tubes are stored in a large specimen tube.

Flow test at 40°C and 45°C was done according to ADA specification No.24 [16]. The amount of force applied to the specimen is 2 kg (19.6 N) force; this load is applied vertically to the specimen by using the standard Vicat apparatus with special modification (Figure 2).

Linear Thermal Expansion was done according to ADA specification No. 24, 2003[16]. The mold was made from glass then duplicated to Aluminum (Figure 3) to prepare the sample. The materials melted to (75 ± 5) °C by using pan and water bath. The melted wax is then poured into a mold that has been lubricated with separating medium (separating film for acrylic resin).The mold was preheated to (55 ±5) ºC. The Aluminum cover preheated to (55 ± 5) º C was placed on the top of the mold, then a weight of 90N (9kg) is placed on the top of the mold for 30 min, after that the weight and cover were removed and excess wax trimmed away and the specimen was stored at room temperature (20 ± 2) ºC for 24 hours before testing. The specimen is heated to 25° C and 40°C and the distance between the reference marks at the lower temperature and change in length on heating to higher temperature is determined by electronic digital caliper (Figure 4).

Microscopical Examination of Waxes: One milligram of wax is placed on glass slide and heated over electric cooker until melting, then a cover slip is placed over it, the cover slip is moved over the glass slide to scatter the wax particle and produce a very thin layer of wax over the glass slide. The glass slide is stored at room temperature until the wax is hardened, then examined with light microscope using the oil lens and magnification (X=400).

Measuring the Accuracy of Experimental Modelling Wax The samples are prepared according to ADA specification No. 24 (6 mm height and 10 mm diameter) cylindrical in shape.

The accuracy is measured by calculating the change in height of samples after 1 hr and 24 hr at room temperature (20±2)°C, and done by taking four measurements around the circumference and one measurement in the center of sample using electronic digital caliper. The mean of five measurements was calculated for each sample. The change is measured as a percentage of the initial height of the sample.

RESULTS and DISCUSSIONS: The results obtained were subjected to statistical analyses in order to determine the best experimental modelling waxes in groups (2 & 3). The results were obtained after the addition of coloring agents.

***Minimum and Maximum Melting Point (Melting range)*:** Descriptive statistics of minimum and maximum melting point of control and groups (1, 2 & 3) are listed in Table (II). The results showed that all the tested samples had a melting range rather than a sharp melting point. These results are in agreement with Craig [19] who revealed that the waxes have a melting range rather than melting point, this can be explained on the basis that the waxes are consist of similar types of molecules of different molecular weights and may contain several types of molecules each having a range of molecular weight.

Analysis of variance ANOVA (Table III) showed that there was a significant difference ($p<0.001$) in the mean value of minimum and maximum melting point of tested samples of control 1 (Polywax™), control 2 (Major®) and experimental groups (2 & 3). Duncan's multiple range test, Figures (5 and 6) showed highest minimum and maximum melting point of the experimental modeling wax 17 than others. The experimental modeling waxes 10, 11, 14, and 15 had the most nearest maximum melting point to control (1 & 2) than others and statistically there was no significant difference between these waxes and control (1 & 2).

The addition of 20% Na-CMC, 20 %Gum Arabic and 20% rosin to hard paraffin as in experimental modelling waxes 20, 21 & 22, led to raise the minimum melting point of the hard paraffin with slight decrease in the maximum melting point. This can be explained as these additives act as a thickener and binder agents and lead to support the mixture during a rise in temperature [20-22].

***Flow at 40ºC and 45ºC*:** The number of samples means and standard deviation, of flow percentages at 40ºC and 45ºC of the tested samples of control, groups 1, 2 & 3 are listed in Table (IV). All experimental modelling waxes had flow properties that coincide with ADA specification No.24; all flow values were located between 50% and 90% at 45ºC which are considered as Type II dental modelling waxes [16].

Analysis of variance (ANOVA), Table (V), show that there was a significant differences ($p< 0.001$) in the mean value of flow percentage at 40ºC and 45ºC of the tested samples of control 1 (Polywax™), control 2 (Major®), and experimental groups

(2 & 3). The results revealed that the soft paraffin had the highest flow percentage than other experimental waxes and this may be related to its low melting point and when the temperature raise to 40ºC and 45ºC, this temperature is near its melting point. This is in agreement with Craig and Powers [23] who stated that the flow greatly increases as the melting point of the wax is approached. The flow reduced more with increasing the percentage of addition from 10% to 20% bees wax and this is in agreement with Craig *et al.*[24], who stated that the addition of beeswax to paraffin leads to raise the transition temperature slightly and thus reducing the flow.

The addition of 10% starch did not affect the flow of hard paraffin significantly as in experimental modelling wax code (16) but when the percentage of addition increased to 20% as in experimental modelling wax code (19) led to reduce the flow of hard paraffin at 40ºC and 45ºC. Also, the addition of 20% Na-CMC, 20 % Gum Arabic, and 20 % rosin to hard paraffin showed the same results. This may be explained on the basis that these materials act as a thickening and binding agents and lead to increase the hardness of paraffin wax thus reducing the flow [20, 25, 26].

The addition of rosin to paraffin produces the highest flow reduction than other additives; this is due to the fact that resins are commonly added to paraffin to produce harder material [19].

Thermal expansion: Descriptive statistics of thermal expansion values of tested samples of control and experimental groups 1, 2, & 3 are listed in Table (VI). The result revealed that all experimental modelling waxes had thermal expansion coincides with ADA specification No. 24, because their expansion not exceeding 0.8% on heating from 25ºC to 40ºC [16].

Analysis of variance (ANOVA), Table (VII), show that there was a significant difference ($p < 0.001$) in the mean value of thermal expansion of tested samples of control 1 (Polywax™), control 2 (Major®), and experimental groups (2 & 3).

Duncan's multiple range test, Figure (7), show that the experimental modelling waxes 10 and 17 had the most nearest thermal expansion percentage to control (1&2) than others. Statistically, there was no significant difference between experimental

modeling wax 10 and control (2). The experimental modelling wax 9 has the highest thermal expansion percentage than others. The experimental modelling waxes 10 (80% hp + 20% bw), 17 (90% bw + 10% starch) and 21(80% hp + 20% gum Arabic) had the most nearest thermal expansion value to the control.

The results showed that the addition of 20% gum Arabic or rosin or Na-CMC to hard paraffin led to reduction in the thermal expansion of hard paraffin. While the addition of 20% starch to hard paraffin led to increase thermal expansion of hard paraffin. This can be explained as the starch will interfere with the secondary valence forces of hard paraffin and when the wax heated, the hydrocarbon chains become more free to move, rotate and expand[(4)] .

Microscopical Examination: Figure (8) showed that the morphology of wax crystals in hard and soft paraffin was varying from tiny needles, elongated needles and plates like crystals. This is in agreement with many authors [(23,27)]. The morphology of natural beeswax crystals was varying from plate, spindle, or leaf like crystals. The experimental modelling wax 10 (80% hp + 20% bw) showed tiny or elongated needle like crystals under Light Microscope using the oil lens and magnification (x=400). These crystals resembling to that seen in contro1 1(Polywax^{TM}) and control 2 (Major®). The experimental modelling wax 17 (90% beeswax + 10% starch) showed plate, or spindle like crystals but not so clear as that seen in natural beeswax and this may be due to the effect of starch that leads to coat the beeswax crystals.

Accuracy of experimental modeling waxes: The results of this study showed that the dimensional changes of paraffin waxes were significantly higher than that of beeswax (Table VIII). These results are in agreement with Sykora and Sutow[(28)] who reported that the little flow at room temperature is often associated with higher wax melting temperature and this has the potential to give less distortion. The addition of 20 % starch, 20% Na-CMC, 20 % Rosin and 20 % gum Arabic to hard paraffin led to reduce the dimensional changes after 1hr and 24 hr. This may be due to the binding properties of these materials that lead to reduce the flow at room temperature and reduce distortion [(20, 21, 22)]. While the addition of beeswax (10% or 20%) to hard

paraffin produced slight decrease in dimensional changes. This may result from increasing the transition temperature and hardness of the hard paraffin: beeswax mixture and reducing the flow at room temperature [24].

Figures (9 and 10) showed that the experimental modelling waxes 7 (90% hp + 10% sp) and 9 (80% hp + 20% sp) had the highest dimensional changes after 1 hr and 24 hr than other experimental waxes. This may be due to weak secondary valence forces between crystals of paraffin waxes [4].

Conclusions: It was concluded that the experimental modelling waxes: (80% hard paraffin + 20% beeswax) and (90% beeswax + 10% starch) have the most nearest properties to control and ADA specification No. 24 than other waxes.

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Table (I): The control with experimental groups and their codes.

Control		**Pure waxes- Group 1**		**Natural waxes mixtures- Group 2**		**(Natural waxes + additives) mixtures - Group 3**	
Materials	**Code**	**Materials**	**Code**	**Materials**	**Code**	**Materials**	**Code**
Polywax™	1	Hard paraffin	3	90% hp + 10% sp	7	90% hp + 10 % starch	16
Major®	2	Soft paraffin	4	90% hp + 10% bw	8	90% bw + 10% starch	17
		Commercial beeswax	5	80% hp + 20 % sp	9	90% sp + 10 % starch	18

Natural beeswax	6	80% hp + 20 % bw	10	80% hp + 20% starch	19
		80% bw + 20% hp	11	80% hp + 20% Na-CMC	20
		80% bw + 20% sp	12	80% hp + 20% gum Arabic	21
		80% sp + 20% bw	13	80% hp + 20 % rosin	22
		80% hp + 15% sp + 5% bw	14		
		70 % hp + 20 % sp + 10 % bw	15		

Hp: hard paraffin, bw: beeswax, sp: soft paraffin, Na-CMC: Sodium carboxymethylcellulose

Table (II) : Descriptive statistics for minimum and maximum melting point of control and groups 1, 2 & 3.

Group	**Code**	**Minimum Melting point(°C)**			**Maximum Melting Point(°C)**		
		N	**Mean**	**SD**	**N**	**Mean**	**SD**
Control	1	3	57.67	0.58	3	64.00	0.00
	2	3	57.33	0.58	3	64.00	0.00
Group 1	3	3	58.67	0.58	3	66.67	0.58
	4	3	43.33	0.58	3	50.00	0.00
	5	3	64.67	0.58	3	71.33	1.15
	6	3	64.33	0.58	3	70.00	0.00
Group 2	7	3	57.33	0.58	3	61.00	0.00
	8	3	59.00	0.00	3	62.67	0.58
	9	3	56.00	0.00	3	61.67	0.58

	10	3	60.00	0.00	3	64.67	0.58
	11	3	58.00	0.00	3	64.33	0.58
	12	3	56.33	0.58	3	60.00	0.00
	13	3	46.00	0.00	3	52.33	0.58
	14	3	57.33	0.58	3	64.00	0.00
	15	3	57.33	0.58	3	64.00	0.00
Group 3	16	3	58.33	0.58	3	67.00	0.00
	17	3	62.67	0.58	3	70.00	0.00
	18	3	43.00	0.00	3	50.67	1.16
	19	3	57.67	0.58	3	67.00	0.00
	20	3	59.00	0.00	3	65.67	0.58
	21	3	60.00	0.00	3	65.33	0.58
	22	3	61.00	0.00	3	66.33	0.58

N: number of samples SD: standard deviation

Table (III): Analysis of variance for minimum and maximum melting points

Minimum melting point					
Control (1) and experimental groups (2 &3).	SS	df	MSE	F	p-value
Between groups	1188.706	16	74.294	473.625	<0.001
Within groups	5.333	34	0.157	.	.
Total	1194.039	50	.	.	.
Control (2) and experimental groups (2 & 3).	SS	Df	MSE	F	p-value
Between groups	1187.412	16	74.213	473.109	<0.001
Within groups	5.333	34	0.157	.	.
Total	1192.745	50	.	.	.

Maximum melting point					
Control (1) and experimental groups (2&3).	SS	df	MSE	F	p-value
Between groups	1178.980	16	73.686	313.167	<0.001
Within groups	8.0	34	0.235	.	.
Total	1186.980	50	.	.	.
control (2) and experimental groups (2&3).	SS	df	MSE	F	p-value
Between groups	1178.980	16	73.686	313.167	<0.001
Within groups	8.0	34	0.235	.	.
Total	1186.980	50	.	.	.

SS: Sum of square df: degree of freedom MSE: mean square

Table (IV): Descriptive statistics for flow at 40 ºC and 45ºC of control, groups 1, 2 & 3.

Group	Flow at 40 ºC (%)			Flow at 45ºC (%)		
	N	Mean	SD	N	Mean	SD
Control 1	3	72.58	0.32	3	83.81	0.292
2	3	55.82	0.672	3	75.20	0.125
Group 1						
3	3	49.85	0.38	3	68.98	0.13
4	3	94.53	0.45	3	95.19	0.04
5	3	66.89	0.3	3	78.75	0.53
6	3	67.51	0.43	3	80.12	0.1

Group 2						
7	3	61.51	0.196	3	72.32	0.23
8	3	47.36	0.099	3	64.67	0.07
9	3	70.10	0.175	3	80.61	0.423
10	3	45.45	0.199	3	63.98	0.225
11	3	47.77	0.419	3	75.74	0.074
12	3	69.62	0.365	3	85.43	0.21
14	3	66.45	0.067	3	76.07	0.298
15	3	66.32	0.261	3	76.03	0.252
Group 3						
16	3	49.21	0.635	3	67.76	0.047
17	3	56.70	0.456	3	74.48	0.025
19	3	47.65	0.455	3	62.23	0.143
20	3	46.12	0.177	3	64.04	0.030
21	3	43.62	0.095	3	61.59	0.306
22	3	42.44	0.120	3	61.45	0.089

SD: standard deviation, N: number of samples

Table (V): Analysis of variance for flow at 40°C, 45°C of the control (1),(2) and experimental groups (2 & 3).

Flow at 40 °C of control (1)					
	SS	df	MSE	F	p-value
Between groups	5119.81	14	365.701	3723.87	<0.001
Within groups	2.946	30	0.0982	.	.
Total	5122.756	44	.	.	.
Flow at 40 °C of the control (2)					
Between groups	4191.507	14	299.393	2463.65	<0.001

Within groups	3.646	30	0.122	.	.
Total	4195.153	44	.	.	.
Flow at 45 °C of control (1)					
Between groups	2865.33	14	204.666	4386.55	<0.001
Within groups	1.4	30	0.04666	.	.
Total	2866.73	44	.	.	.
Flow at 45 °C of control (2)					
Between groups	2428.875	14	173.491	4128.56	<0.001
Within groups	1.261	30	0.04202	.	.
Total	2430.136	44	.	.	.

SS: Sum of square df: degree of freedom MSE: mean square

Table (VI): Descriptive statistics for thermal expansion of control and experimental groups 1, 2 & 3.

Thermal expansion (%)							
Control Group		Group 1		Group 2		Group 3	
Code	**Mean±SD**	**Code**	**Mean±SD**	**Code**	**Mean±SD**	**Code**	**Mean±SD**
1	0.27± 0.003	3	0.34±0.01	7	0.47±0.007	16	0.36±0.001
2	0.23±0.001	4	0.40±0.00	8	0.37±0.002	17	0.27±0.001
		5	0.29±0.00	9	0.49±0.001	19	0.41±0.006
		6	0.29±0.01	10	0.22±0.002	20	0.31±0.003
				11	0.34±0.002	21	0.28±0.003
				12	0.37±0.005	22	0.29±0.002
				14	0.32±0.006		
				15	0.34±0.003		

SD: standard deviation, For each subgroup 3samples

Table (VII): Analysis of variance for thermal expansion of control (1), (2) and experimental groups (2 & 3).

	SS	df	MSE	F	p-value
Thermal expansion of control (1)					
Between groups	0.233	14	0.01661	1242.5	<0.001
Within groups	0.0004	30	0.000013	.	.
Total	0.233	44	.	.	.
Thermal expansion of control (2)					
Between groups	0.256	14	0.01829	1411.66	<0.001
Within groups	0.00039	30	0.000013	.	.
Total	0.256	44	.	.	.

SS: Sum of square df: degree of freedom MSE: mean square

Table (VIII): Descriptive statistics for accuracy after 1 hr and 24 hr of control, groups 1, 2, & 3.

Group		Dimensional changes after 1 hr (%)			Dimensional changes after 24 hr (%)		
		N	**Mean**	**SD**	**N**	**Mean**	**SD**
Control	1	3	0.33	0.003	3	0.33	0.002
	2	3	0.33	0.003	3	0.34	0.002
				Group 1			
3		3	0.35	0.000	3	0.35	0.00
4		3	0.41	0.000	3	0.41	0.00
5		3	0.13	0.000	3	0.32	0.00
6		3	0.14	0.000	3	0.33	0.00
				Group 2			
7		3	0.44	0.002	3	0.49	0.001
8		3	0.32	0.002	3	0.32	0.002
9		3	0.37	0.002	3	0.43	0.003
10		3	0.32	0.002	3	0.32	0.002
11		3	0.31	0.001	3	0.31	0.001
12		3	0.19	0.003	3	0.32	0.001
14		3	0.32	0.002	3	0.36	0.001

15	3	0.23	0.002	3	0.24	0.001
			Group 3			
16	3	0.33	0.001	3	0.42	0.001
17	3	0.17	0.002	3	0.17	0.001
19	3	0.33	0.002	3	0.33	0.001
20	3	0.24	0.001	3	0.33	0.001
21	3	0.17	0.002	3	0.34	0.001
22	3	0.16	0.001	3	0.16	0.001

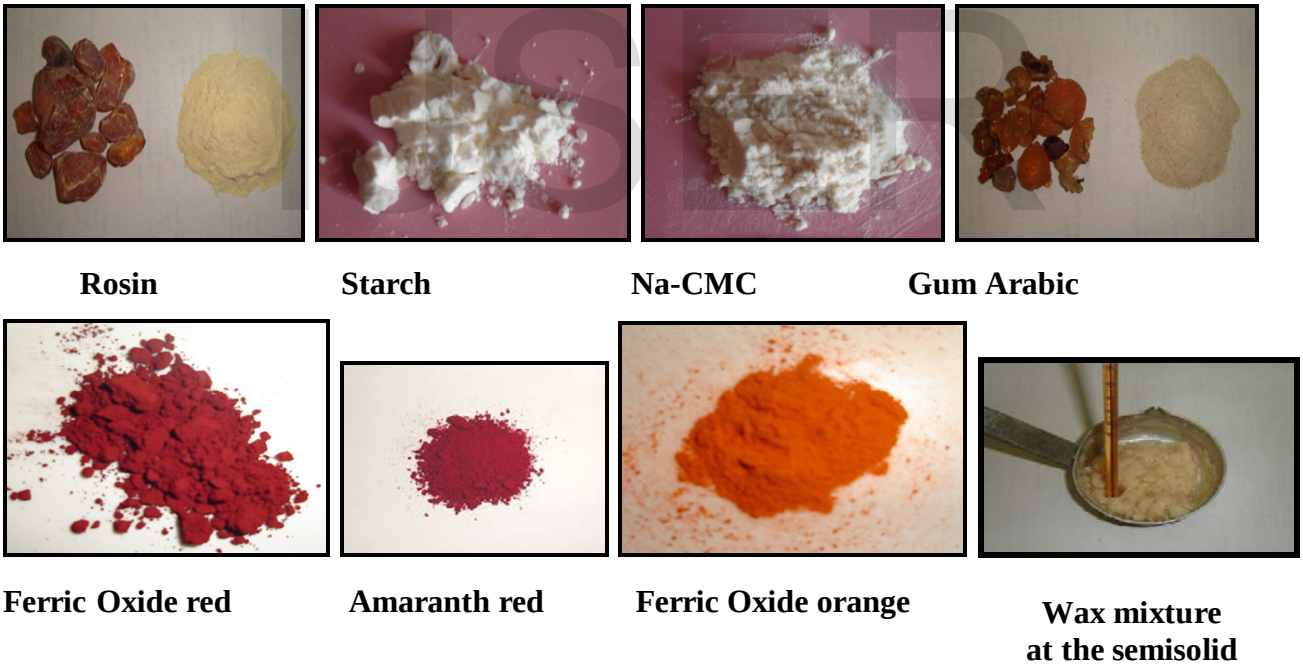


Figure (1): Additives materials used in this study and Wax mixture at the semisolid.

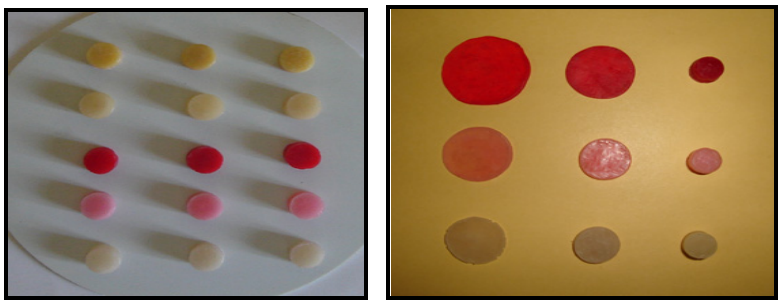


Figure: (2) Different wax flow at (20 ± 2)ºC, 40 °C and 45° C from right to left side.

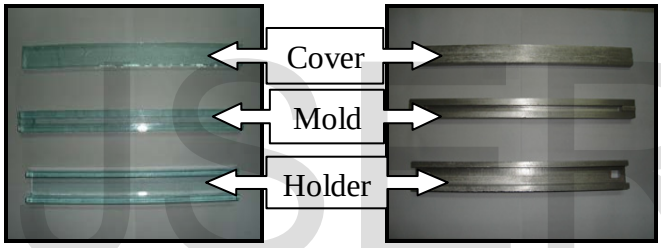


Figure 3: Mold for thermal expansion test made from glass and Aluminum.

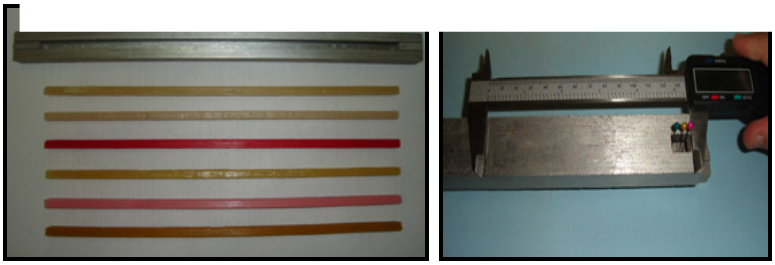


Figure 4: Samples of different waxes placed under the holder of the thermal expansion test.

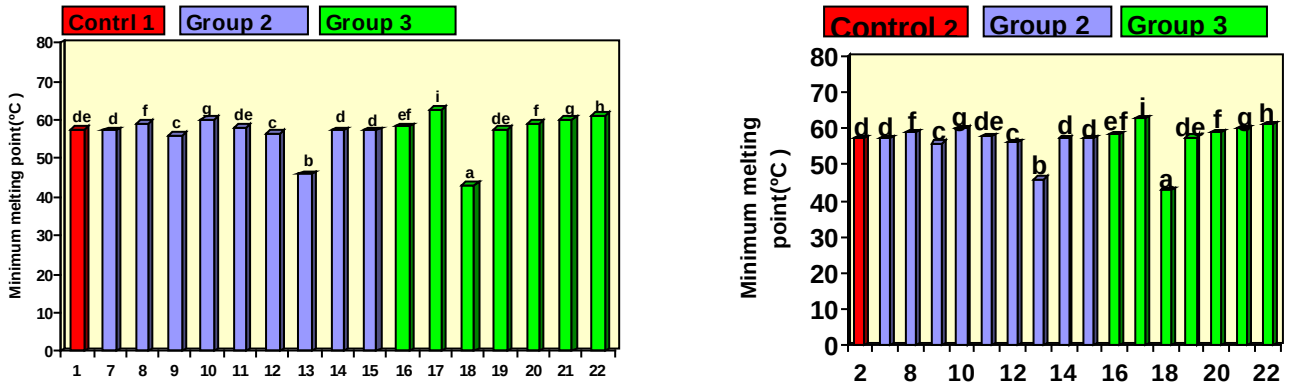


Figure (5): Duncan's multiple range test of the minimum melting point of the controls (1) and (2) in relation to experimental modelling waxes in groups 2 & 3.

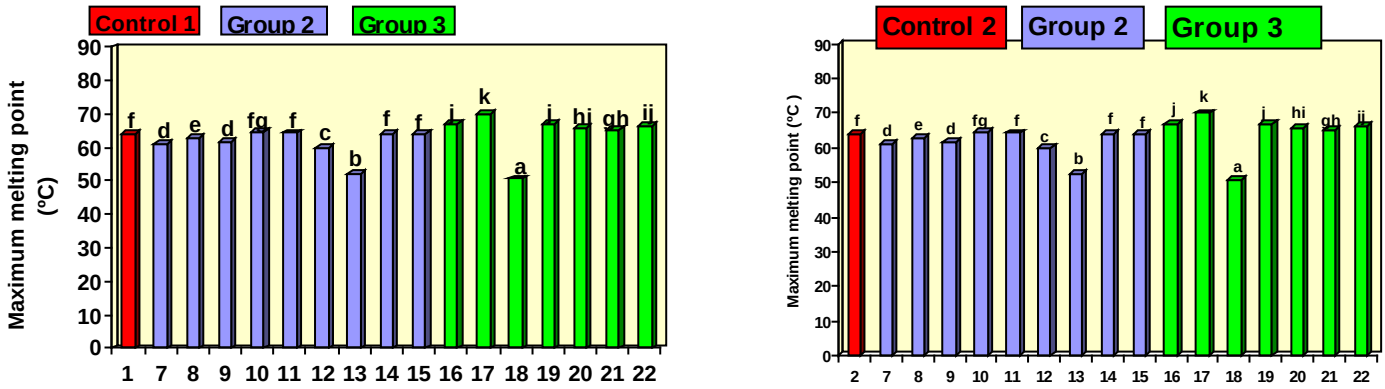


Figure (6): Duncan's multiple range test of the maximum melting point of the controls (1) and (2) in relation to experimental modelling waxes in groups 2 & 3.

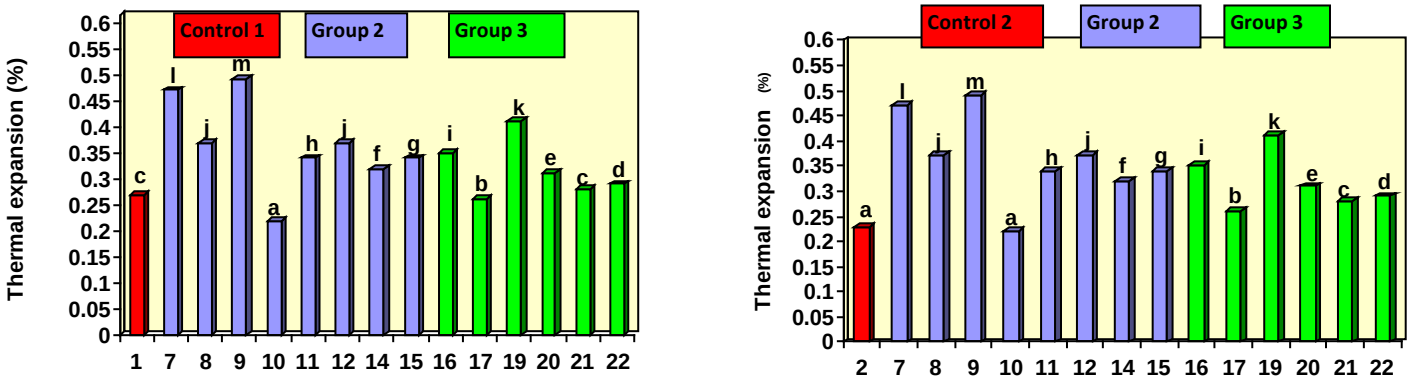
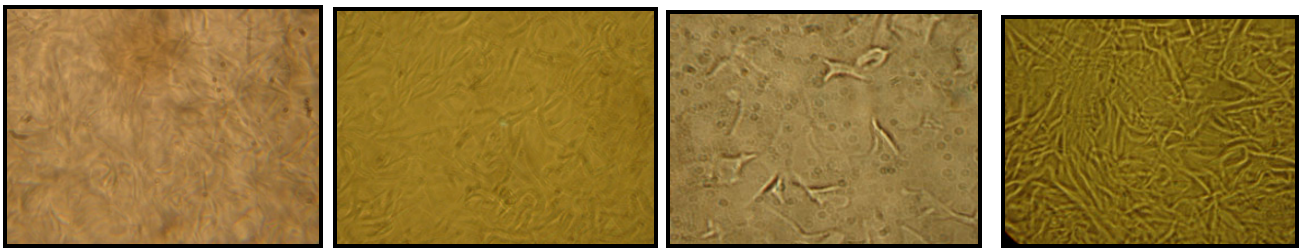
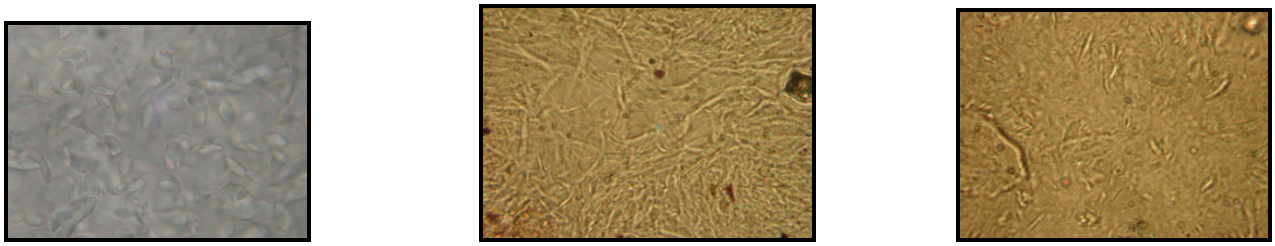


Figure (7): Duncan's multiple range test of thermal expansion of controls (1) and (2) in relation to experimental modelling waxes in groups 2 & 3.



Polywax™ (1) Major® (2) Hard paraffin (3) Soft paraffin (4)



Natural Beeswax (6) **80% Hard paraffin +20%Beeswax (10)** **90% Beeswax+10% Starch(17)**

Figure (8): Microscopical examination of wax materials

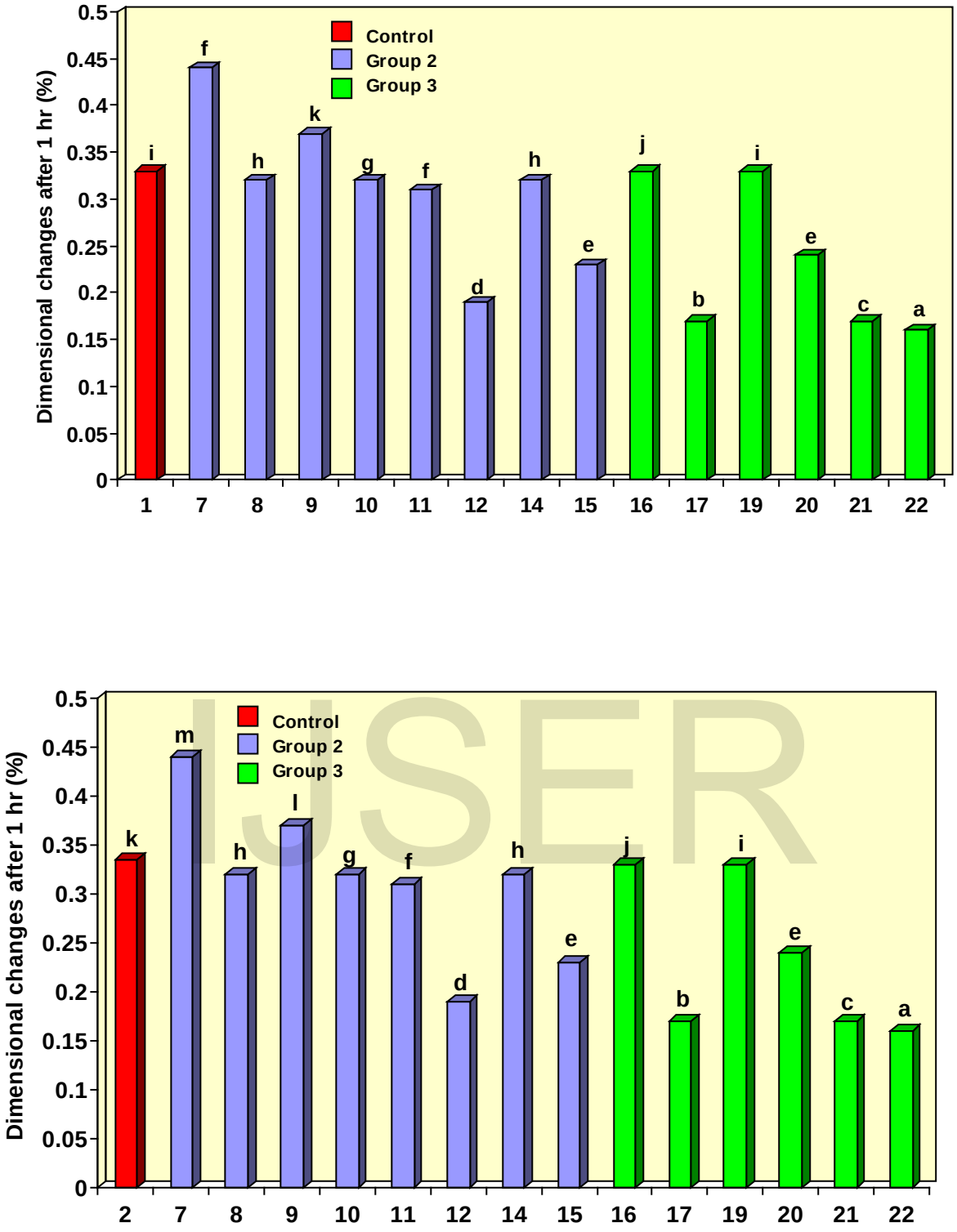


Figure (9): Duncan's multiple range test of accuracy after 1 hr of the controls (1) and (2) in relation to experimental modelling waxes in groups 2 & 3.

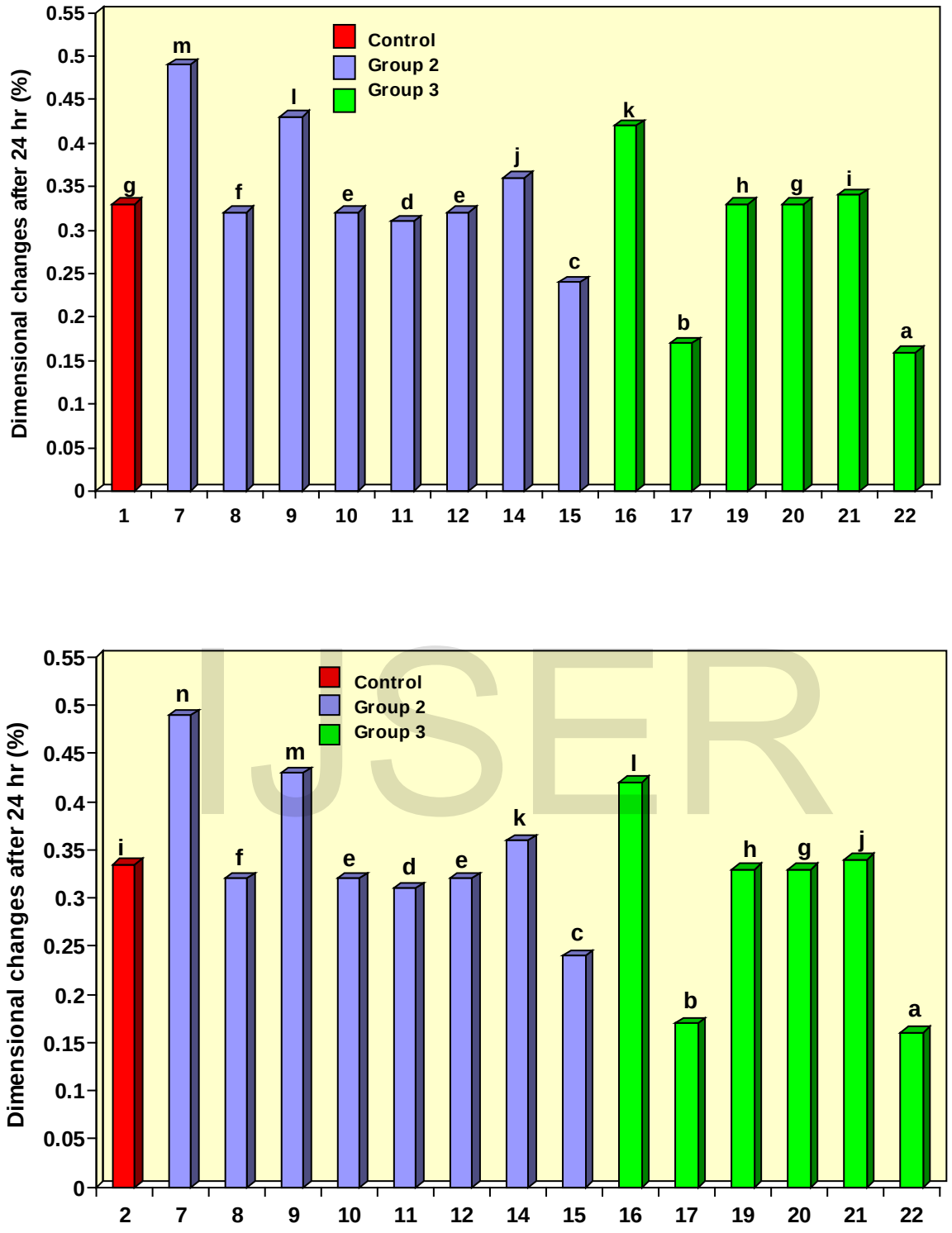


Figure (10): Duncan's multiple range test of accuracy after 24 hr of the controls (1) and (2) in relation to experimental modelling waxes in groups 2 & 3.