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Studying the Functional Properties of Linen Fabrics Treated with Microwave and Natural Green Dyes

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Abstract: The suitable functional properties of linen fabrics which dyed by natural dyes was realized and its dye ability was improved by treatment with microwave radiation. The most suitable criteria (weave structure, natural dyes concentration, treatment time by microwave radiation, type of green natural dyes) of linen fabric were determined. The used fabrics have the following specifications: (Linen), three weave structures (Twill weaves, Imitation-gauzu weaves, Honey-comb weaves), weft density 12 picks/cm, warp yarn and weft yarn 100 % flax fiber No.30/2 (English numbering). The fabrics were treated with Arkofix resin (100 g/L). The resin was fixed on fabrics by microwave radiation (510 watts) at three interval times (30 sec., 1.0 min., 1.5 min.). The linen fabrics were dyed with spinach and parsley green dyes at concentration of 5%, 10% for each dye. The produced linen fabrics were evaluated by measuring various factors affecting on fastness properties and the crease recovering. The results were statistically analyzed using Analysis of Variance to obtain correlation and multi-regression equations. The multi-pronged Radar chart (multi-axis) was used to evaluate quality of clothes produced under investigation. The results reveal that the weave structure (Honey-comb weaves) after treatment with Arkofix-resin, microwave radiation (1.5 min.) and dyed with spinach dye of 10% concentration is the best for all performances by factor of quality 712.22. The least sample produced under investigation was the weave structure (Twill weaves) before treatment with parsl dye of 10% concentration by factor of quality 47.

Keywords: Linen Fabrics; Natural Dyes; Microwave Radiation, Functional Properties.

1. Introduction:

Linen, which is a natural bast fibre, has unparalleled characteristics such as a feel of freshness and a magnificent brilliance. It is very hygienic and imparts an air of satisfaction and style to the wearer¹. In spite of its highly functions and features, it has weak resistance to wrinkle. This is a characteristics of large defects², requiring subjected, processing to improve the efficiency of the textile product and suitable for manufacturing of clothes with quality specifications³. In the field of textile industry, we note increasing in pollution levels due to using synthetic dyes and this has an adverse effect on human health⁴. So, there is an increase on the demand on Eco-Textiles in most export markets. These days and future⁵, due to increased awareness of the environmental markets prompt scientific research returning to natural dyes to reduce environmental pollution and preservation of human life⁶. Due to the requirements of environmental protection laws, the use of the microwave radiation in the treatment of cellulosic fabrics to improve their physical properties and increase the effectiveness of the dyeing process is a means of biotechnology to ensure the achievement of the so-called clean production⁷.

The functional properties of linen fabrics dyed with natural dyes (madder, cochineal, turmeric, catechu, saffron, henna and indigo) were investigated⁸. Other natural dyes (turmeric, madder, tea and Bailasan Mecca were used for dyeing knitted fabrics, cotton and linen fabrics and their functional properties were also investigated⁹⁻¹⁰. Nigar Merdan and others used microwave and lactase enzyme to improve the functional

properties of cellulose fiber¹¹. Many other studies on using microwave radiation and/or natural dyes to improve the functional properties of different textile were observed in literature¹²⁻¹⁴. Mustafa Tutak and others used natural dyes (madder root, henna, buckthorn and walnut shell) to improve the functional properties of linen fabrics¹⁵.

This study aimed to improve the functional properties of linen fabrics dyed with natural dyes as well as improve the efficiency of the dyeing process using microwave radiation in order to reach the most appropriate of the following:

- 1. Weave structure achieve the best functional properties of the produced linen fabrics under investigation.
- 2. Treatment time of microwave radiation.
- 3. The type of green natural dye achieves the best functional properties of the produced linen fabrics.
- 4. A suitable concentration of the natural dye achieves the highest efficiency for the dyeing process of the produced linen fabrics.

The research work based on the following hypotheses:

- 1. There are statistically significant differences between the weave structure and functional properties of the produced linen fabrics under research.
- 2. There are statistically significant differences between treatment time of microwave radiation and functional properties of linen fabrics.
- 3. There are statistically significant differences between the type of natural green dye and functional properties of the produced linen fabrics.
- 4. There are statistically significant differences between the concentration of the natural dye and functional properties of the produced linen fabrics.

2. Experimental and laboratory tests:

2.1. Materials:

The used linen fabric is produced in Sharkiya Company for Spinning and Weaving, Zagazig, Egypt and has the following specifications:

- 100% linen, indirectly numbering 30/2 and Spun by wet spinning, 12 picks/cm.
- Weave structures: Twill weaves, Honey-comb weaves, Imitation-gauzu weaves.

The produced fabrics underwent initial treatments to remove bush, boiling in alkaline solution, bleaching and treatment with Arkofix-resin material (Eco-friendly, free from formaldehyde, obtained from Clariant Swiss Co.). Treatment with Arkofix gives fabrics with crease recovery and improvement of fabrics properties at a concentration of 100 g/L. The treatment with Arkofix-resin was fixed with microwave radiation at three interval times (30 s, 1 min., 1.5 min.) and a constant power of 510 watts.

2.2. Steps of treatment with Arkofix material:

- (i) The concentrations of Arkofix, magnesium chloride (catalyst to complete the reaction) and lummen (Lubricating material to soften the cloth surface) are 100, 15 and 2 g/L, respectively.
- (ii) Era step: the era of fabrics was done after treatment in the presence of Arkofix and other materials.
- (iii) Drying step: drying fabrics in air.
- (iv) Fixation step: Arkofix material was fixed inside the produced linen fabrics using microwave radiation at three interval times (30 sec, 1 min., 1.5 mins.) and constant power of 510 watts.

2.3. Extraction of Natural Dyes:

Two types of natural green dyes are extracted from a plant source (parsley and spinach). Copper sulfate was also used as a kind of metal fasteners (Mordant) in the fixation process of colors on fabrics. The green leaves of spinach or parsley were washed thoroughly with water and then dried well from washing water. The

drying of parsley and spinach plants was done in the shade, separately. The parsley and spinach plants were grinded, separately to be turned into powder. The leaf powders of parsley and spinach were sieved through fine sieves. The powders were placed in round flasks, and then added ethanol to each flask (100 gm powder/1.25 L ethanol). The mixtures were left for 48 hours (soaking) with continuous stirring daily then filtration using Wattman filter paper (No. 1). Repeat these steps of soaking the filtered powders in ethanol and the filtrations were three times. Sum the filtrate of each plant and dry it in hot water bath (40 °C) using Rotary Evaporator under reduced pressure. The green dye of each plant was obtained as paste then re-dissolved in water or the suitable solvent to prepare a stock solution of 50 % concentration and suitable for further dilutions. The dyes concentration percent were confirmed using UV-Visible Spectrophotometer.

Dying process:

Samples which will be dyed were weighted. Dyeing bath which contains the dye solution by the required quantity (1 g of dye material: 40 ml of water) were prepared. Then, a dye concentration of 5% and once again 10% were added to dyeing bath. Copper sulfate (5 gm/L) was dissolved in water and added to the dyeing bath. The cloth samples were immersed in the well prepared dyeing bath and stirred. The mixture was heated to 100 °C for 1 *h* with continuous stirring then, gradually cooled. The linen fabric samples were removed from dyeing bath, washed and finally dried at ambient conditions. Testing and analysis to naturally dyed linen fabrics were carried out to find the relationships between different variables using Applied Statistics. These tests are crease recovering testing, light fastness, wash fastness, rubbing fastness, iron fastness and color strength.

3. Results and Discussion

3.1. Effect of various factors on the functional properties of the linen fabrics produced under investigation:

Analysis of variance (ANOVA) was carried out to study the effect of different factors including weave structure, treatment time by microwave radiation, type of the used dye and dye concentration on the crease recovering testing, light fastness, wash fastness, rubbing fastness, iron fastness and color strength of the dyed linen fabrics produced under investigation.

3.1.1. Studying of various factors on the rubbing fastness (dry, wet):

The studying factors have a non-significant impact on the property for rubbing fastness (dry). But all studying factors have a significant effect on the rubbing fastness (wet) with the exception of the dye concentration which has a non-significant effect on rubbing fastness (wet), see Table 1.

Table 1. Analysis of variance (N-Way ANOVA) for studying the impact of various factors on the rubbing fastness (dry, wet)

Rubbing	Source	Type III Sum of Squares	df	Mean Square	F	Sig.
	Weave structure	76.781	2	38.391	1.109	0.340
	Treatment time	131.458	3	43.819	1.266	0.299
Dwy	Dyes concentration	36.750	1	36.750	1.062	0.309
Dry	Type of dyes	18.750	1	18.750	0.542	0.466
	Error	1384.260	40	34.607		
	Corrected Total	1648.000	47			
	Weave structure	0.698	2	0.349	2.978	0.042
	Treatment time	4.932	3	1.644	14.030	0.000
	Dyes	0.047	1	0.047	0.400	0.531
Wet	concentration					
	Type of dyes 4.380		1	4.380	37.378	0.000
	Error	4.688	40	0.117		
	Corrected Total	14.745	47			

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(i) The multi-linear regression equation for the rubbing fastness (dry) is as follows:

$$Y = 3.51 + 1.26 X_1 + 1.30 X_2 + 1.75 X_3 - 1.25 X_4 \qquad R^2 = 0.$$

Where: Y = rubbing fastness (dry), X_1 = weave structure, X_2 = treatment time, X_3 = dye concentration, X_4 = the type of dye. It represents an inverse correlation between various factors of study and the rubbing fastness (dry). Fig. 1 shows that the weave structure (Honey-comb weaves), treatment time at 1.5 minutes by microwave radiation and 10% concentration of the parsley dye achieve higher stability of the rubbing fastness (dry).

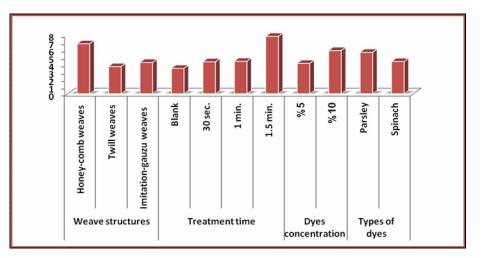


Fig. 1. Effect of various factors of study on the rubbing fastness (dry).

(ii) The multi-linear regression equation for the rubbing fastness (wet) is as follows:

$$Y = 1.89 + 0.031 X_1 + 0.221 X_2 + 0.063 X_3 - 0.604 X_4$$
 R² = 0.68

It represents a direct relationship between various factors of study and the rubbing fastness (wet). Fig. 2 shows that the weave structure (Imitation-gauzu weaves), processing time at 1 minute by microwave radiation and 10% concentration of the spinach dye achieve the highest constancy of rubbing fastness (wet) for fabrics.

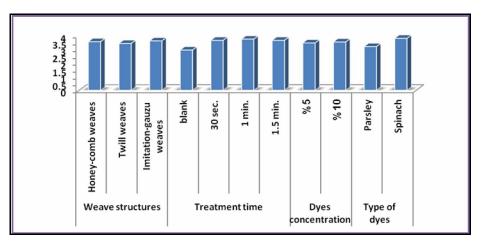


Fig. 2. Effect of various factors of study on the rubbing fastness (wet).

3.1.2. Studying of various factors on wash fastness:

Both of the treatment time and the type of dye have a significant effect on wash fastness, while weave structures and dye concentration have a non-significant effect on wash fastness for linen fabrics, see Table 2.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Weave structure	0.031	2	0.016	0.132	0.876
Treatment time	11.458	3	3.819	32.377	0.000
Dyes concentration	0.021	1	0.021	0.177	0.677
Type of dyes	1.021	1	1.021	8.653	0.005
Error	4.719	40	0.118		
Corrected Total	17.250	47			

Table 2. Analysis of variance (N-Way ANOVA) for studying the impact of various factors on the wash fastness

The multi-linear regression equation for the wash fastness is as follows:

$$Y = 2.13 + 0.16 X_1 + 0.333 X_2 - 0.042 X_3 + 0.292 X_4 \qquad R^2 = 0.72$$

It represents a direct relationship between various factors of study and the wash fastness. Fig. 3 shows that the weave structure (Twill weaves), treatment time at 1.0 minute by microwave radiation and 5 % concentration of the spinach dye achieve higher stability of the wash fastness.

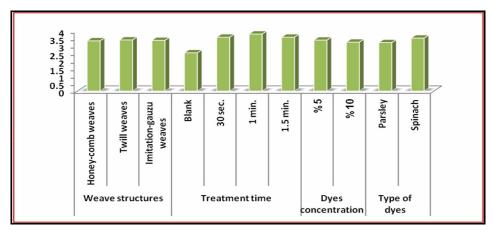


Fig 3. Effect of various factors of study on the wash fastness.

3.1.3. Studying of various factors on the iron fastness:

All factors have a non-significant effect on iron fastness, see Table 3.

The multi-linear regression equation for the iron fastness is as follows:

 $Y = 7.75 - 0.938 X_1 - 0.467 X_2 + 1.33 X_3 - 1.332 X_4 \qquad R^2 = 0.11$

It represents an inverse relationship between various factors of study and the iron fastness. Fig. 4 shows that the weave structure (Honey-comb weaves), treatment time at 1.0 minute by microwave radiation and 10% concentration of the parsley dye achieve higher stability to iron fastness.

Table 3: Analysis of variance (N - Way ANOVA) for studying the impact of various factors on the iron fastness

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Weave structure	38.135	2	19.068	0.912	0.410
Treatment time	29.167	3	9.722	0.465	0.708
Dyes concentration	21.333	1	21.333	1.021	0.318
Type of dyes	21.333	1	21.333	1.021	0.318
Error	835.948	40	20.899		
Corrected Total	945.917	47			

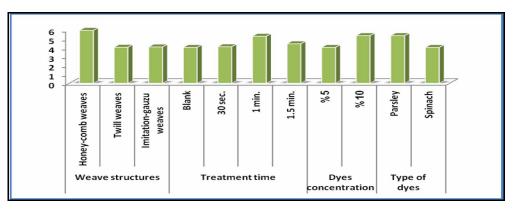


Fig. 4. Effect of various factors of study on the iron fastness.

3.1.4. Studying of various factors on the light fastness:

All factors have a significant effect on light fastness, but the weave structure has a non-significant effect on the light fastness, see Table 4.

Table 4. Analysis of variance (N-Way ANOVA) for studying the impact of various factors on the light
fastness

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Weave structure	0.031	2	0.016	0.154	0.858
Treatment time	9.125	3	3.042	30.026	0.000
Dyes concentration	0.521	1	0.521	5.141	0.029
Type of dyes	1.021	1	1.021	10.077	0.003
Error	4.052	40	0.101		
Corrected Total	14.750	47			

The multi-linear regression equation for the light fastness is as follows:

$$Y = 2.52 + 0.013 X_1 + 0.367 X_2 - 0.208 X_3 + 0.292 X_4$$
 $R^2 = 0.72$

It represents a direct relationship between various factors of study and the light fastness. Fig. 5 shows that the weave structure (Imitation-gauzu weaves), treatment time at 1.5 minutes by microwave radiation and 5% concentration of the spinach dye achieve higher stability to light fastness.

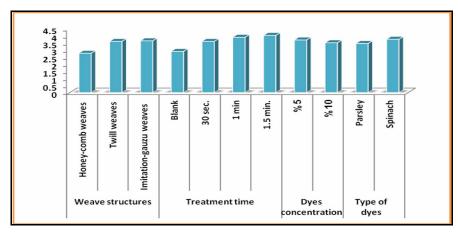


Fig. 5. Effect of various factors of study on the light fastness.

The multi-linear regression equation for the color strength is as follows:

$$Y = 55.79 - 3.18 X_1 + 11.48 X_2 + 14.91 X_3 + 14.58 X_4$$
 R²= 0.65

Where Y= color strength. It represents a direct relationship between various factors of study and the color strength. All factors have a significant effect on the color strength of the naturally dyed linen fabrics produced under investigation except weave structure which has a non-significant effect on color strength, Table 5.

Table 5. Analysis of variance (N-Way ANOVA) for studying the impact of various factors on the color strength

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Weave structure	866.625	2	433.313	2.149	0.130
Treatment time	8990.917	3	2996.972	14.863	0.000
Dyes	2670.083	1	2670.083	13.242	0.001
concentration					
Type of dyes	2552.083	1	2552.083	12.657	0.001
Error	8065.542	40	201.639		
Corrected Total	23145.250	47			

Fig. 6 shows that the weave structure (Honey-comb weaves), treatment time at 1.5 minutes by microwave radiation and 10% concentration of the spinach dye achieve the highest stability of color strength.

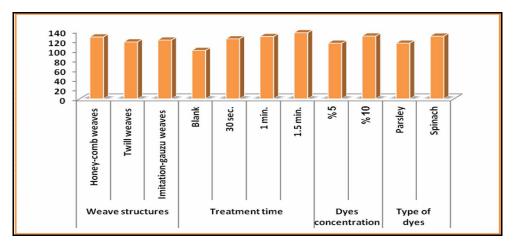


Fig 6. Effect of various factors of study on the color strength

3.3. Studying of various factors on the crease recovery (in the directions of wrap and weft):

All factors of the study have a significant effect on the crease recovery in the wrap direction except for the dye type which has a non-significant effect. But, all factors of study have a significant effect on the crease recovering in the weft direction see Table 6.

Table 6. Analysis of variance (N-Way ANOVA) for studying the impact of various factors on the crease recovery (in the directions of wrap and weft)

Crease recoverin g	Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Wrap	Weave structure	332.375	2	166.187	10.183	0.000
	Treatment time	1441.896	3	480.632	29.449	0.000
	Dyes concentration	346.688	1	346.688	21.242	0.000
	Type of dyes	58.521	1	58.521	3.586	0.066
	Error	652.833	40	16.321		

	Corrected Total	2832.313	47			
	Weave structure	255.167	2	127.583	18.248	0.000
	Treatment time	1114.167	3	371.389	53.119	0.000
Weft	Dyes concentration	208.333	1	208.333	29.797	0.000
	Type of dyes	208.333	1	208.333	29.797	0.000
	Error	279.667	40	6.992		
	Corrected Total	2065.667	47			

The multi-linear regression equation for crease recovery in the wrap direction is as follows:

$$Y = 82.43 + 3.21 X_1 + 4.67 X_2 + 5.37 X_3 - 2.20 X_4; \qquad R^2 = 0.77$$

It represents a direct relationship between various factors of study and the crease recovery in the wrap direction.

The multi-linear regression equation for crease recovery in the weft direction is as follows:

 $Y = 1.89 + 0.031 X_1 + 0.221 X_2 + 0.063 X_3 - 0.604 X_4, \qquad R^2 = 0.88$

It represents a direct relationship between various factors of study and crease recovery in the weft direction. Fig. 7 shows that the weave structure (imitation-gauzu weaves), treatment time at 1.5 minutes by microwave radiation and 10 % concentration of the parsley dye achieve the highest stability to crease recovery in the wrap direction.

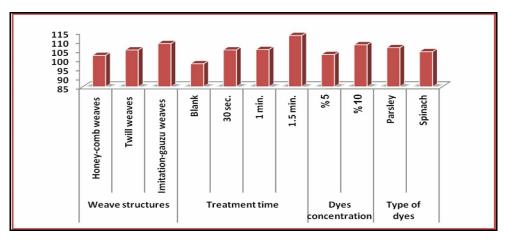


Fig 7. Effect of various factors of study on crease recovery in the wrap direction

Sample number	Weave structure	Treatment time	Dyes conc.	Type of dyes	Rubbing (dry)	Rubbing (wet)	Washing	Iron	Light	Color strength	Crease recovery (warp)	Crease recovery (weft)	Factor of quality		
1	Hanna	blank			60	50	40	70	60	51.02	77.50	82.68	491.20		
2	Honey-	30sce			80	70	60	80	70	64.80	80.83	85.83	591.46		
3	comb weaves	1 min			90	80	80	90	80	58.67	82.50	85.83	647.00		
4	weaves	1.5 min			90	60	60	80	70	56.63	97.50	94.49	608.62		
5		blank			60	50	60	70	60	51.02	76.67	78.74	506.43		
6	Twill	30sce	5%		90	60	80	90	70	58.16	87.50	90.55	626.21		
7	weaves	1 min	370		80	70	70	80	80	56.12	87.50	89.76	613.39		
8		1.5 min			80	60	70	90	90	55.61	95.00	93.70	634.31		
9	T	blank			60	60	50	70	60	51.02	80.83	85.04	516.89		
10	Imitation-	30sce			80	70	60	80	70	60.71	94.17	93.70	608.58		
11	gauzu	1 min			90	70	80	80	70	57.65	84.17	87.40	619.22		
12	weaves	1.5 min		D 1	80	70	70	90	80	59.69	100.00	100.00	649.69		
13		blank		Parsley	70	60	40	70	60	51.02	80.00	84.25	515.27		
14	Honey-	30sce	-		80	70	80	80	70	63.78	83.33	88.19	615.30		
15	comb	1 min	-		80	70	70	80	70	64.29	85.83	90.55	610.67		
16	weaves	1.5 min	-		90	70	70	90	80	64.80	99.17	97.64	661.60		
17		Blank			60	40	40	70	50	51.02	78.33	81.89	471.24		
18	Twill	30sce	1.00/		80	60	70	80	70	64.80	95.00	92.13	611.92		
19	weaves	1 min	10%	-	90	70	80	90	80	65.31	89.17	92.13	656.60		
20		1.5 min	-		80	70	60	90	70	62.76	97.50	95.28	625.53		
21	T 10.01	blank	-		70	40	40	70	50	51.02	82.50	85.83	489.35		
22	Imitation-	30sce				80	70	80	90	60	57.14	96.67	94.49	628.30	
23	gauzu	1 min			80	70	70	80	80	66.33	96.67	93.70	636.69		
24	weaves	1.5 min	-		90	80	70	90	70	65.82	100.00	100.00	665.82		
25	**	blank		Spinach	80	70	60	60	60	51.02	79.17	78.74	538.93		
26	Honey-	30sce			100	80	70	80	70	61.22	80.83	80.31	622.37		
27	comb	1 min			90	80	80	80	70	59.69	81.67	82.68	624.04		
28	weaves	1.5 min		-			90	70	70	90	80	68.88	83.33	84.25	636.46
29		blank			70	80	60	70	70	51.02	81.67	81.89	564.58		
30	Twill	30sce	5%		90	80	70	80	80	55.61	83.33	84.25	623.20		
31	weaves	1 min	5%		80	70	70	90	80	61.73	85.00	86.61	623.35		
32		1.5 min			80	70	80	90	90	71.94	87.50	89.76	659.20		
33	T	blank			70	60	50	70	70	51.02	83.33	82.68	537.03		
34	Imitation-	30sce			100	90	80	80	80	67.86	85.83	85.83	669.52		
35	gauzu	1 min			90	70	80	90	90	61.22	87.50	88.98	657.70		
36	weaves	1.5 min			100	80	80	90	90	64.80	89.17	91.34	685.30		
37	Honey-	blank	10%		80	70	60	70	60	51.02	83.33	82.68	557.03		
38	comb	30sce	1		90	80	70	80	80	82.65	86.67	85.83	655.15		
39	weaves	1 min	1		100	80	80	90	80	94.90	89.17	88.19	702.25		

Table 7. Total quality assessment to functional properties of the investigated linen fabrics

40		1.5 min	90	80	80	90	90	100.00	91.67	90.55	712.22
41		blank	70	60	60	60	50	51.02	85.00	84.25	520.27
42	Twill	30sce	80	70	70	80	70	57.14	87.50	88.19	602.83
43	weaves	1 min	90	80	70	80	70	68.37	90.83	90.55	639.75
44		1.5 min	90	80	80	90	80	78.57	94.17	92.91	685.65
45	Imitation	blank	80	70	50	70	50	51.02	87.50	87.40	545.92
46	Imitation- gauzu	30sce	90	80	70	90	80	65.31	90.00	90.55	655.86
47	weaves	1 min	100	90	80	80	90	74.49	94.17	92.13	700.78
48	weaves	1.5 min	100	90	70	90	80	87.24	95.83	94.49	707.57

Fig. 8 shows that the weave structure (imitation-gauzu weaves), treatment time at 1.5 minutes by microwave radiation and 10 % concentration of the spinach dye achieve the highest stability to crease recovery in the weft direction.

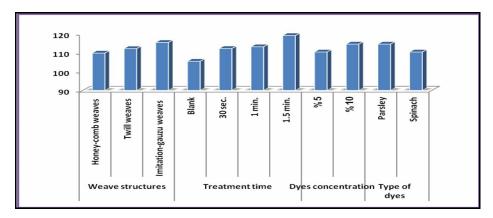


Fig. 8. Effect of various factors of study on crease recovery in the weft direction

3.4. Total quality assessment of the functional properties to linen fabrics:

Quality assessment of the functional properties (weave structure, treatment time by microwave radiation, dye concentration, and type of dye) to linen fabrics was done before and after treatments to find the best one using multi-pronged Radar charts. This method expresses the quality assessment of the investigated linen fabrics through using the following properties (rubbing, light, iron, washing, color strength and the crease recovery). This assessment transfer the mean values of the results of measurements to a relative comparison between 0.0 - 100.0 where the largest comparison value will be the best in all different properties, see Table 7.

The used linen fabrics with Honey-comb weave has the best functional properties and performance after treatment with Arkofix-resin ,microwave radiation for 1.5 minutes, 10 % dye concentration and spinach dye with a quality factor of 712.22, while the less samples obtained before treatment with microwave radiation were found to be Twill weaves, at a 10 % dye concentration and spinach dye with a quality factor of 471.24, Figs. 9,10.

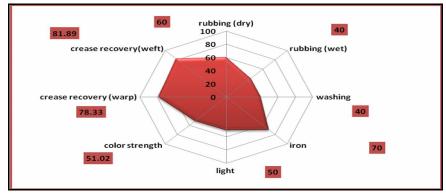


Fig 9. The less produced fabric samples under investigation.

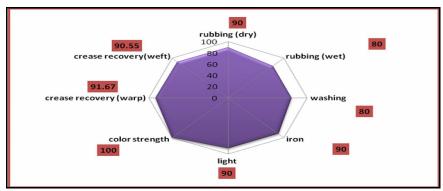


Fig 10. The best produced fabric samples under investigation

Conclusion:

Therefore, the researchers recommend further studies to use many of the natural dyes in this field and replace the industrial dyes that may cause some environmental problems and treatment of ore must also be environmentally friendly. The suitable functional properties of linen fabrics which dyed by natural dyes was realized and its dye ability was improved by treatment with microwave radiation. The used fabrics have the following specifications: (Linen), three weave structures (Twill weaves, Imitation-gauzu weaves, Honey-comb weaves). The fabrics were treated with Arkofix resin (100 g/L). The used linen fabrics were dyed with spinach and parsley green dyes at concentration of 5%, 10% for each dye. The results revealed that the weave structure (Honey-comb weaves) after treatment with Arkofix-resin, microwave radiation (1.5 min.) and dyed with spinach dye of 10% concentration is the best for all performances by factor of quality 712.22.

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