Comparative Study for the Effect of Fixative Material Type and Perfume Formulation Parameters on the Fixation time of Local Formulated Perfume with Brand Perfumes

Dr. Alaa D. Jawad Al-Bayati
Institute of Technology ,Technical Middle University/Baghdad.
Email: bayati.engineer@gmail.com

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ABSTRACT

The effect of type of fixative material and the perfume formulation parameters (Concentration of ethyl alcohol, percentage of perfume and the percentage of the fixative used) were studied to determine the optimum conditions give perfume with high retention time (fixative), also the fixation time was compared with brand perfumes. Four fixative materials were studied; Sandal oil, Musk oil, Glycerin and benzyl benzoate. Design of experiments (DOE) with Minitab 15 software was done and leaded to minimum 20 runs necessary for this study. For the four fixatives the runs were eighty .The maximum fixation time of all the fixation materials are 6.3, 6.1, 5.9 and 5.9 h/g for Glycerin, Musk, Sandalwood and benzyl benzoate respectively with formulation parameters of (75% alcohol concentration, 30% perfume addition and 0.1% fixative material) for Musk, Glycerin and benzyl benzoate fixative materials and (85% alcohol concentration, 17.5% perfume addition and 0.55% fixative material) for Sandalwood as fixative material. The brand perfumes as Channel give 2 h/g fixation time and for many other brand perfumes give fixation time in the range of 2.2-2.9 h/g.

Keywords: perfume, perfume fixatives, perfume formulation, fixative time for perfumes.

دراسة مقارنه تأثير نوع المادة المثبتة وشروط متغيرات تركيب العطور على زمن بقاء العطور المركبة محليا مع العطور الاجنبية المشهورة

الخلاصة

تم دراسة تأثير نوع المادة المثبتة ومتغيرات تركيب العطور (تركيز الكحول الاثيلي المستخدم في التركيبة، نسبة العطر المضاف ونسبة المادة المثبتة المستخدمة) من اجل ايجاد الشروط المثلى التي تعطي العطر زمن بقاء عالي (فترة ثبات للعطر) وكذلك تم مقارنة زمن بقاء العطور التي تم تحضيرها من قبلنا مع زمن بقاء عطور اجنبية ذات علامة تجارية معروفة. لقد تم استخدام أربعة مواد مثبتة : زيت الصندل ، زيت المسك، الكليسرين وبنزوات البنزيل.ان تصميم التجارب بواسطة برنامج مينيتاب Minitab 15 ادى الى ٢٠ تجربة من اجل دراسة المتغيرات اعلاه عند كل نوع من انواع مثبتات العطر Fixative وبما انه تم دراسة ٤ أنواع من المثبتات فقد تم عمل ٨٠ تجربة (٢٠ تجربة لكل نوع) . أظهرت النتائج ان أعظم زمن بقاء للعطر لجميع المواد المثيته المستخدمة هو 6.3، 6.1، 5.9 و 5.9 ساعة/غم للكليسرين ، المسك، صندل الخشب وبنزوات البنزيل على التوالي وكانت شروط التركيب هي (75% تركيز الكحول ، ٣٠% تركيز العطر و0.1% تركيز المادة المثبتة المضافة) عند استخدام المسك والكليسرين وبنزوات البنزيل كمواد مثبته و (85% تركيز الكحول الاثيلي ، 17.5% تركيز العطر و0.55% تركيز المادة المضافة) عند استخدام زيت خشب الصندل كمادة مثبتة. إن العطور الاجنبية ذات الماركة مثل عطر شانيل كان زمن البقاء له 2 ساعة/غم والعديد من الانواع الاخرى العالمية المشهورة كان زمن يقائها يتراوح بين 2.9-2.2 ساعة/ غم .

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2412-0758/University of Technology-Iraq, Baghdad, Iraq

INTRODUCTION

Perfumes are liquid mixture of fragrant components dissolved in a suit'able solvent such as ethyl alcohol. It is normally sprayed on the skin or clothes in order to get the fragrance smell through its vaporization and the vapors will reach the surroundings. So vaporization of the perfume is an indication of the existence of the perfume and the rate of vaporization means the rate of getting the fragrance materials found in the perfume liquid solution. Impact, tenacity, diffusion and volume are important performance factors for the perfume. Tenacity is an indication of the lasting of the perfume after its application, it measures the persistence of a fragrance for long time near the evaporation source [1].Perfumes are formulated by adding essential oils (concentrates) incorporated with ethanol and water and some fixatives. The concentration of the essential oils added depended on the type of the perfume made as shown in table (1) [2]. Tenacity is the gradual release or constant volatility of the perfume for long period of time. Many substances are used as fixatives to give the good tenacity of the perfume [3].

Evaporation of the fragrance materials in the perfume product is important [4]. Fixatives is added to perfume solutions in order to obviate the difficulty of a series impressions of odor in the perfume because of different volatilities in the notes of the perfume, fixatives are substances of high boiling points and it will retard the rate of evaporation of the fragrances materials in the perfume, the fixatives give the perfume long time of lasting. There are different types of fixatives used in the formulation of perfumes such as; essential oils, animal secretions, synthetic chemicals and resinous materials [5, 6, 7, 8, 9, 10]. Ethanol can be assumed as a partially oxidized hydrocarbon [11]. So it is of interest to study the effect of different fixatives on the rate of evaporation of perfume formula made and to find the time of lasting to each fixative and the effect of the concentration of the mobile solvent (ethanol), the concentration of the essential oil on each type of fixative used. Four fixatives were used in the study, Sandalwood, Musk, and Glycerin and benzyl benzoate.

Table (1): Percent of essential oil according to type of perfume

	Type	Essential oil %
1	Perfume	15-25
2	Eau de Perfume	8-15
3	Eau de toilitte	4-10
4	Eau de cologne	2-5

Materials and Methods

Experiments were designed to study the effect of three parameters which are; concentrations of ethyl alcohol, concentration of perfume material and the weight percentage of the fixative used on the fixation time of the perfume formulated. Using MiniTab 15 Software leads to 20 runs of experiment as shown in table (2). The minimum and maximum concentrations for the studied variables are shown in table (3). The runs were executed with four types of fixative which are the Sandalwood, Musk, and Glycerin and benzyl alcohol i.e. (80) eighty runs were done. The type of essential oils used is Aramis and Christiane Dior which was purchased from the local markets in Baghdad. Ethyl alcohol which was used is 95% purity type and it was diluted with distilled water to get the concentration of (75-95%) ethyl alcohol as the runs appeared in the design of experiment.

Table (4) show the abbreviations of the fixatives and essential oils (perfumes) used in our study. Laboratory balance with accuracy of (4) digits after the decimal point was used (Sartorius).

Materials

The materials used are:
1. Essential oils (Christian Dior).
2. Ethyl alcohol of 95% concentration.
3. Fixative materials (Sandal wood, Glycerin, Benzyl benzoate and Musk oil).
4. Laboratory balance with 4 digits after the decimal point (Sartorius).

Method

- The perfumes were prepared according to the formula obtained by the design of experiment shown in table (2), the formula used with each of the four fixative materials studied.
- One gram of each of the prepared perfumes was put in a watch glass.
- The watch glass was put on the laboratory balance and the time registered as zero minute.
- The weight of the perfume stayed on the watch glass was registered each (5-10) minute for a period of 1-2 hours.
- The rate of evaporation of the perfume put was calculated for each run (i.e. perfume formula) as follows:

Rate of evaporation of the perfume (g/h) = (Weight of the perfume at time 1 – Weight of the perfume at time 2)/ (Time 2-Time1). [$\frac{2 - W}{}$.

-The time of fixation of the perfume (h/g) was calculated from the rate of evaporation of the perfume (1/time rate of evaporation).
-An Excel program was designed to find the rate of evaporation (fixation time).

Table (2): Design of experiments

Run NO.	Sample Name	conc.of alcohol%	conc.of perfume %	conc. of fixatives %
1	P1 F1 - 1	85	17.5	0.55
2	P1 F1 - 2	75	5	0.1
3	P1 F1 - 3	85	17.5	0.55
4	P1 F1 - 4	95	30	0.1
5	P1 F1 - 5	95	5	0.1
6	P1 F1 - 6	75	5	1
7	P1 F1 - 7	68	17.5	0.55
8	P1 F1 - 8	75	30	1
9	P1 F1 - 9	95	5	1
10	P1 F1 - 10	85	17.5	0.55
11	P1 F1 - 11	85	17.5	0.55
12	P1 F1 - 12	95	30	1
13	P1 F1 - 13	85	17.5	0.55
14	P1 F1 - 14	85	17.5	1.3
15	P1 F1 - 15	85	3.5	0.55
16	P1 F1 - 16	85	38.5	0.55
17	P1 F1 - 17	85	17.5	0.2
18	P1 F1 - 18	95	17.5	0.55
19	P1 F1 - 19	75	30	0.1
20	P1 F1 - 20	85	17.5	0.55

Table (3): Minimum and maximum values for the studied variables

Name of material	Code
Sandal Fixative	F1
Musk Fixative	F2

Glycerin	F3
Benzyl Alcohol	F4
Christian Dior Perfume	P1

Table (4): Codes of fixative materials and essential oils (perfumes) used in the study

No.	Variable	Minimum %	Maximum %
1	Volumetric percentage of essential oil added	5	30
2	Concentration of ethyl alcohol	75	95
3	Volumetric percentage of Fixative materials added	0.1	1

Results

- The results shown in table (5), explain that the fixation time depends on the type of fixative material and the perfume formula parameters. (Concentration of alcohol and perfume and concentration of fixative material). The maximum fixation time was obtained with formula parameters shown in table (6) below.

Table (5) the results of the Fixation time

Run No.	Sample name	Fixation time (h/g)	Run No.	Sample name	Fixation time (h/g)
1	P1F1-1	3.5	41	P1F1-11	5.8
2	P1F2-1	3.2	42	P1F2-11	5.2
3	P1F3-1	4.2	43	P1F3-11	5.2
4	P1F4-1	4.2	44	P1F4-11	4.6
5	P1F1-2	3.7	45	P1F1-12	2.3
6	P1F2-2	3.7	46	P1F2-12	2.8
7	P1F3-2	4	47	P1F3-12	2.9
8	P1F4-2	3.4	48	P1F4-12	2.9
9	P1F1-3	2.9	49	P1F1-13	5.8
10	P1F2-3	2.9	50	P1F2-13	5.2
11	P1F3-3	2.8	51	P1F3-13	5.2
12	P1F4-3	2.8	52	P1F4-13	4.6
13	P1F1-4	3.2	53	P1F1-14	4.2
14	P1F2-4	3.1	54	P1F2-14	4.5
15	P1F3-4	2.6	55	P1F3-14	4.1
16	P1F4-4	3.4	56	P1F4-14	4.3
17	P1F1-5	3.2	57	P1F1-15	3.1
18	P1F2-5	3	58	P1F2-15	3.3
19	P1F3-5	2.9	59	P1F3-15	3.2
20	P1F4-5	2.6	60	P1F4-15	3.4
21	P1F1-6	5	61	P1F1-16	4.1
22	P1F2-6	4.6	62	P1F2-16	4
23	P1F3-6	2.5	63	P1F3-16	3.9
24	P1F4-6	2.7	64	P1F4-16	3.9
25	P1F1-7	5	65	P1F1-17	4
26	P1F2-7	5	66	P1F2-17	4.2
27	P1F3-7	5.1	67	P1F3-17	4
28	P1F4-7	5.2	68	P1F4-17	4.6
29	P1F1-8	5.1	69	P1F1-18	5
30	P1F2-8	4.8	70	P1F2-18	5.6

31	P1F3-8	5.1	71	P1F3-18	5.5
32	P1F4-8	4.9	72	P1F4-18	5.2
33	P1F1-9	5.2	73	P1F1-19	2.9
34	P1F2-9	4.6	74	P1F2-19	6.1
35	P1F3-9	4.7	75	P1F3-19	6.3
36	P1F4-9	4.4	76	P1F4-19	5.9
37	P1F1-10	5.9	77	P1F1-20	5.9
38	P1F2-10	5.2	78	P1F2-20	5.2
39	P1F3-10	5.2	79	P1F3-20	5.2
40	P1F4-10	4.6	80	P1F4-20	4.6

- The formulas which lead to the maximum time of fixation with each fixative material and its formulation conditions are shown in table (6).It was shown that the perfume formulation which gave maximum fixation time for Musk oil, Glycerin and Benzyl benzoate fixatives are 75% concentration of ethyl alcohol, 30% essential oil and 0.1% concentration of fixative material. While for Sandal oil fixative the perfume formula which give the maximum fixation time was 85% concentration of ethyl alcohol, 17.5% essential oil and 0.55% concentration of fixative material. This means that the Sandal wood fixative perfume formula of maximum fixation time is more economic because it uses less concentration of essential oil which is normally the expensive material.

Table (6) the conditions of perfume formulation which give maximum fixation time

No.	Type of Fixative	Max. time of Fixation h/g	Conc. of ethyl Alcohol %	% essential oil	% of fixative material
1	Sandal oil	5.9	85	17.5	0.55
2	Musk oil	6.1	75	30	0.1
3	Glycerin	6.3	75	30	0.1
4	Benzyl benzoate	5.9	75	30	0.1

- Table (7) show the fixation time for some of foreign brand perfumes.

Table (7) Fixation time for foreign perfumes

No.	Foreign Perfume Name	Fixative Time h/g
1	Channel	2
2	English Perfume	2.2
3	Cascade	2.9
4	Escade	2.7
5	Elle	2.4

- The effects of fixative material on the fixation time for each fixative concentration used, is shown in Figure (1). The figure shows that for the concentration of 0.1% fixative material, Sandal wood fixative was not good fixation time while the concentration of 0.5% fixative material, the Sandal wood fixative material is the best for getting maximum fixation time, while for the other fixatives studied is not good.

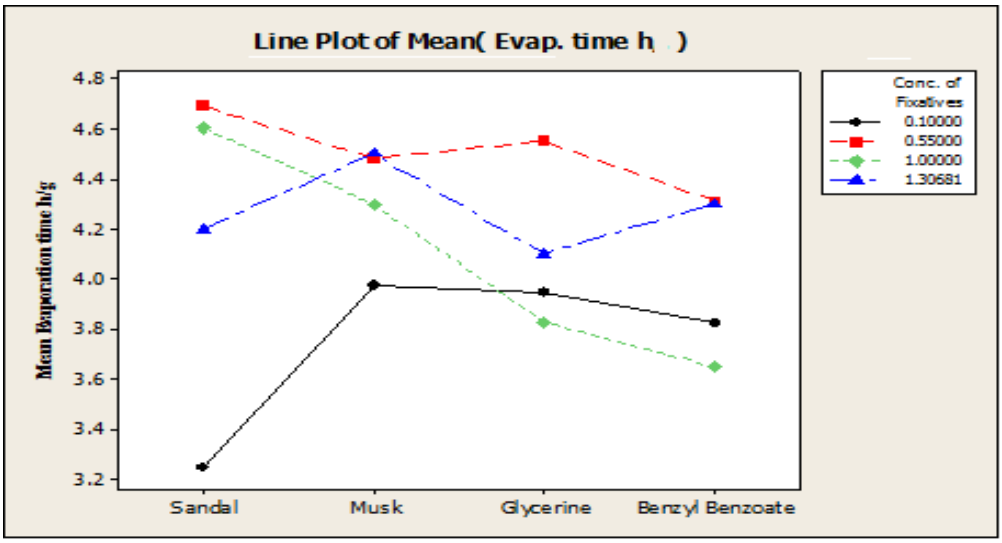


Figure (1): The effects of fixative concentration on the fixation time for each fixative material used.

- Contour plots for the fixation time with the conditions of formulation variables for each fixative material can be seen in Figures (2-5). These figures give us the range of perfume formulation variables of each fixative material which can be used for each range of fixation time required for the perfume.

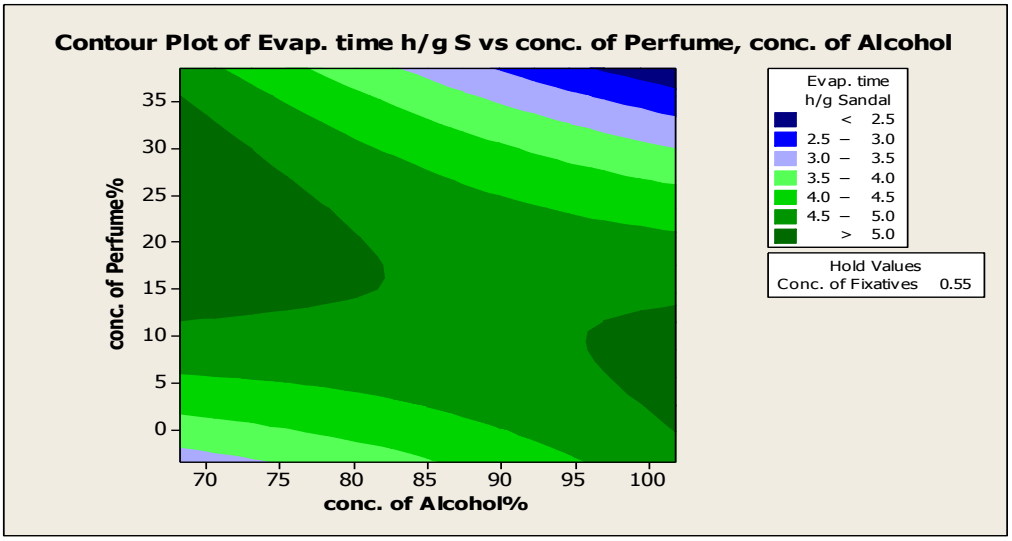


Figure (2) The contour plot of total evaporation time versus concentration of perfume and concentration of Sandal fixative

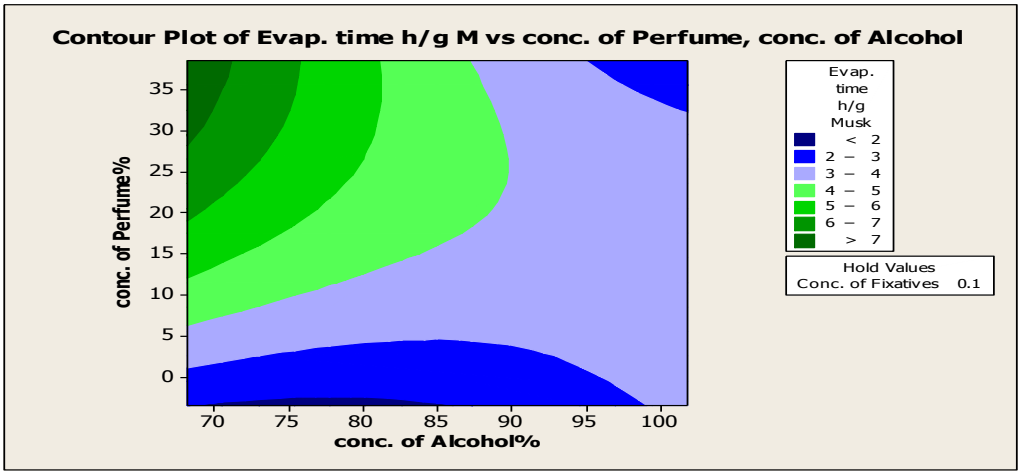


Figure (3) The contour plot of total evaporation time versus concentration of perfume and concentration of Musk fixative

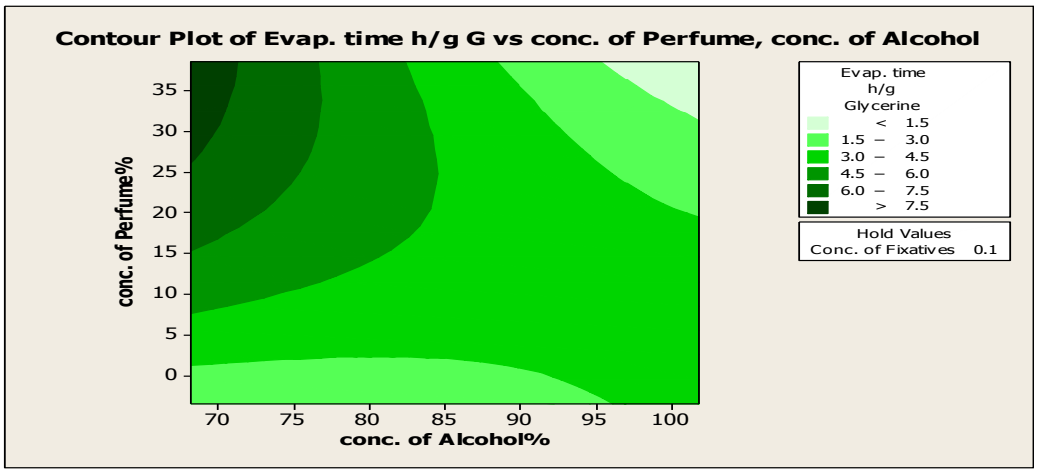


Figure (4) The contour plot of total evaporation time versus concentration of perfume and concentration of Glycerin fixative.

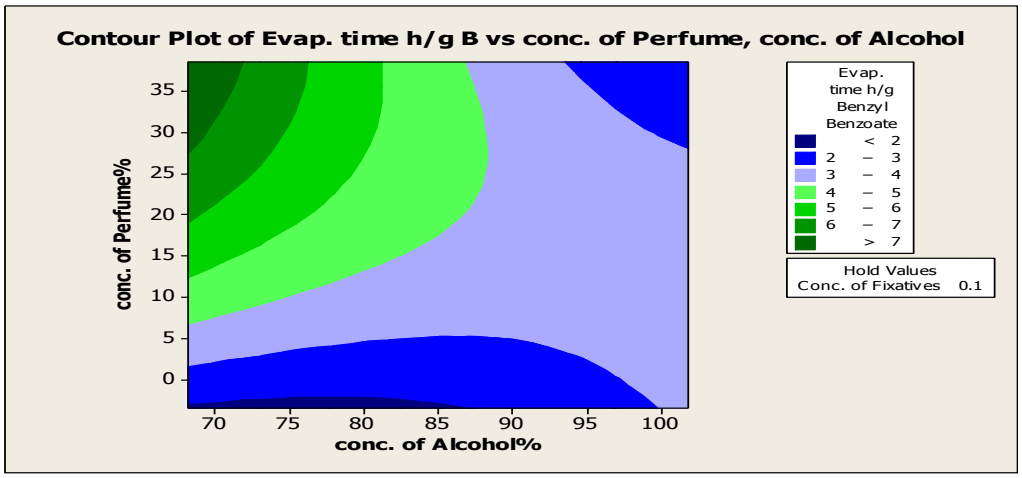


Figure (5) The contour plot of total evaporation time versus concentration of perfume and concentration of benzyl benzoate fixative.

- Figures (6-9) show the main effects of perfume formulation variables studied on the evaporation time (the fixation time) of the perfume. For Sandal fixative material, it was shown that the concentration of fixative of 0.55%, concentration of perfume of 17.5% and 85% ethyl alcohol concentration. The fixation time increased with increasing the concentration of the essential oils and decreased with increasing the concentration of ethyl alcohols for each of the fixatives used, this was due to the high boiling point of the essential oils and the low boiling point of the ethyl alcohol.

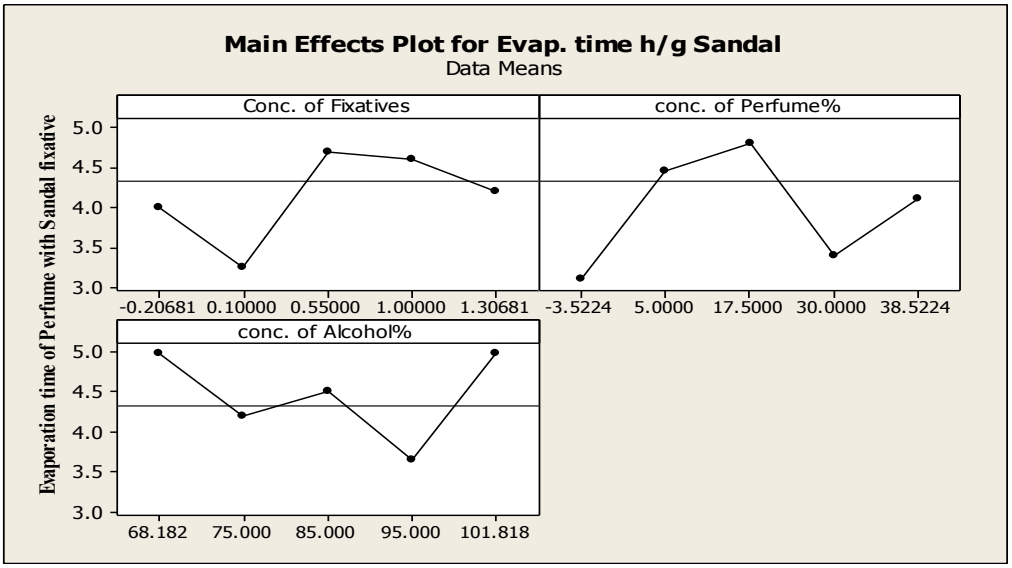


Figure (6) the main effects of the perfume formulation variables studied on the quality of evaporation rate (Time of Fixation) for the perfume formulated with Sandal fixative.

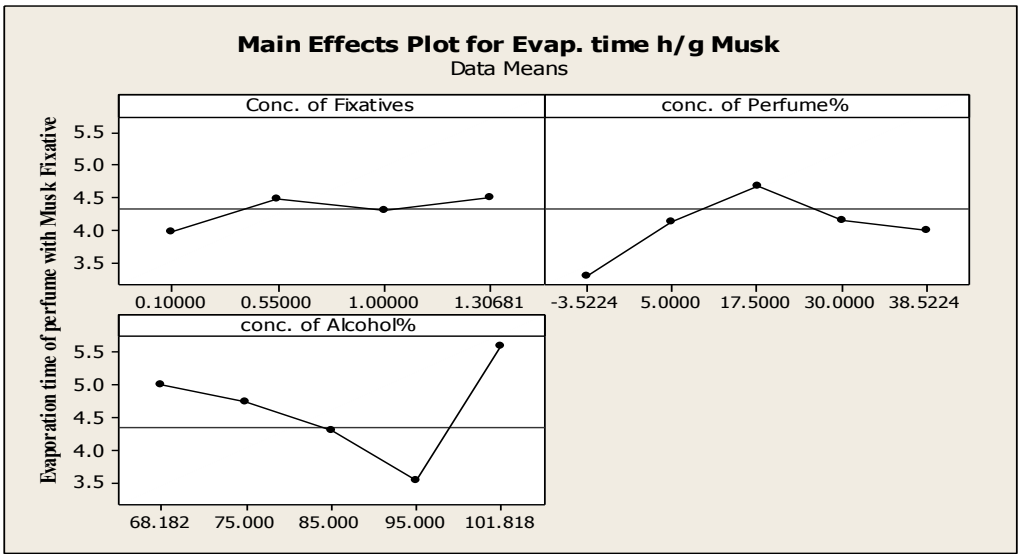
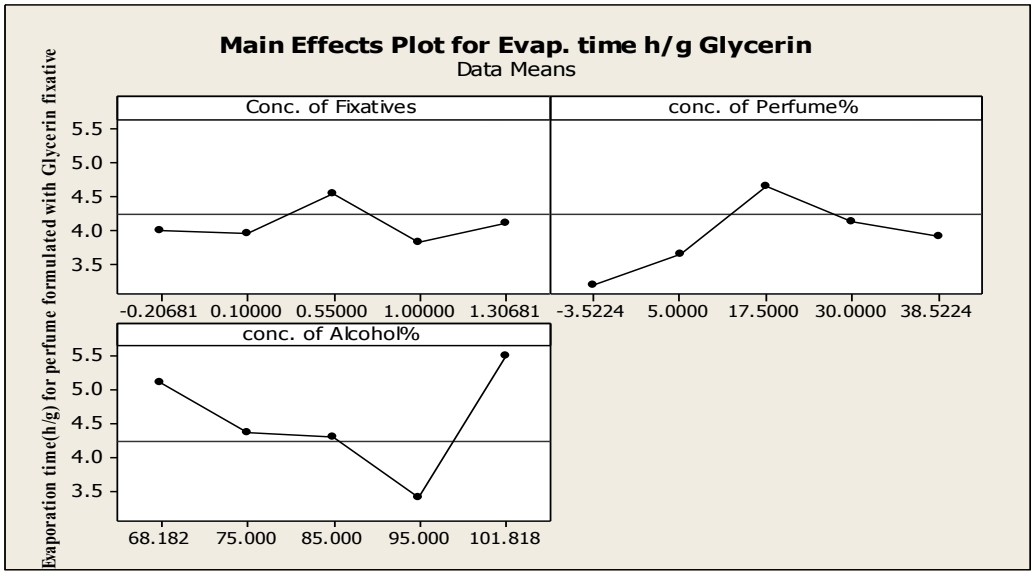
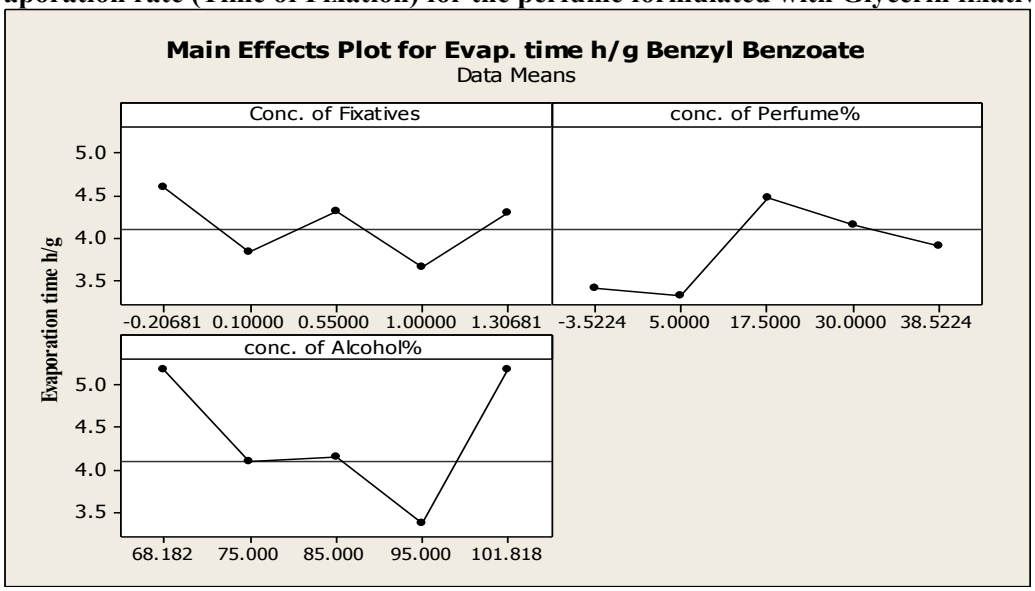


Figure (7) the main effects of the perfume formulation variables studied on the quality of evaporation rate (Time of Fixation) for the perfume formulated with Musk fixative.



Figures (8) the main effects of the perfume formulation variables studied on the quality of evaporation rate (Time of Fixation) for the perfume formulated with Glycerin fixative.



Figures (9) the main effects of the perfume formulation variables studied on the quality of evaporation rate (Time of Fixation) for the perfume formulated with Benzyl Benzoate fixative.

Model Fit for the Evaporation rate of the perfumes:

-Regression Analysis gave us the evap. rate versus Conc. of Fix, conc. of Perfumes formulated with Musk (F1), Sandal (F2), Glycerin (F3) and Benzyl Benzoate fixatives (F4) as shown below:

-evap. rateF1 (MusK) = 0.097 - 0.0398 Conc. of Fixatives + 0.00109 conc. of Perfume% + 0.00186 conc. of Alcohol%

-evap. rate F2 (Sandal) = 0.050 - 0.0195 Conc. of Fixatives - 0.00052 conc. of Perfume% + 0.00246 conc. of Alcohol%

- evap. rate F3 (Glycerine) = 0.117 + 0.0023 Conc. of Fixatives - 0.00140 conc. of Perfume% + 0.00189 conc. of Alcohol%

- **evap.rate F4 (Benzyl Benzoate)** = 0.159 + 0.0058 Conc. of Fixatives - 0.00185 conc. of Perfume%+ 0.00146 conc. of Alcohol%

Figures (10-13) shows the interaction effects of the evaporation rate of different fixative materials.

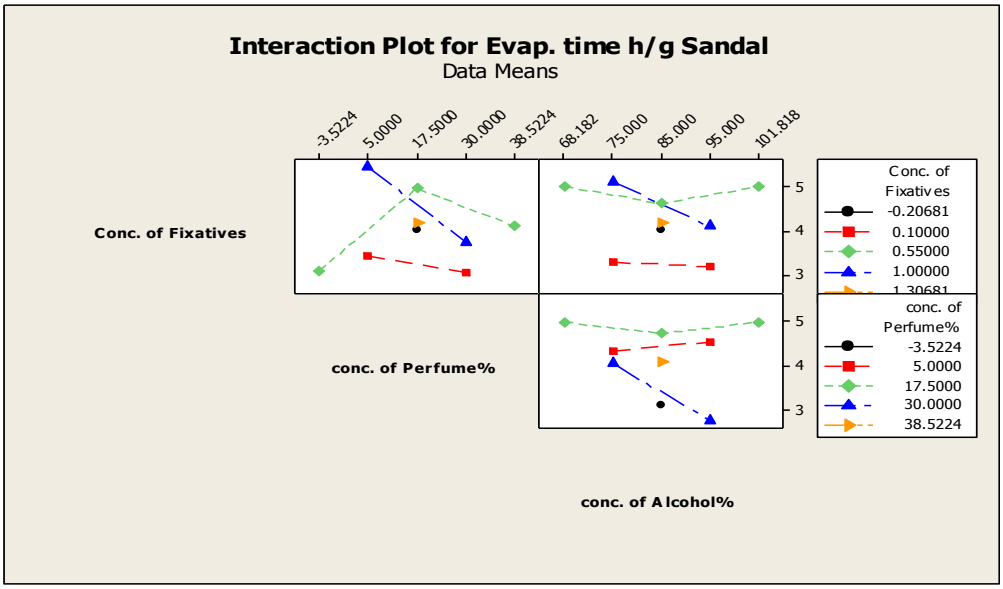


Figure (10) the interaction effect of the evaporation rate with Sandal fixative.

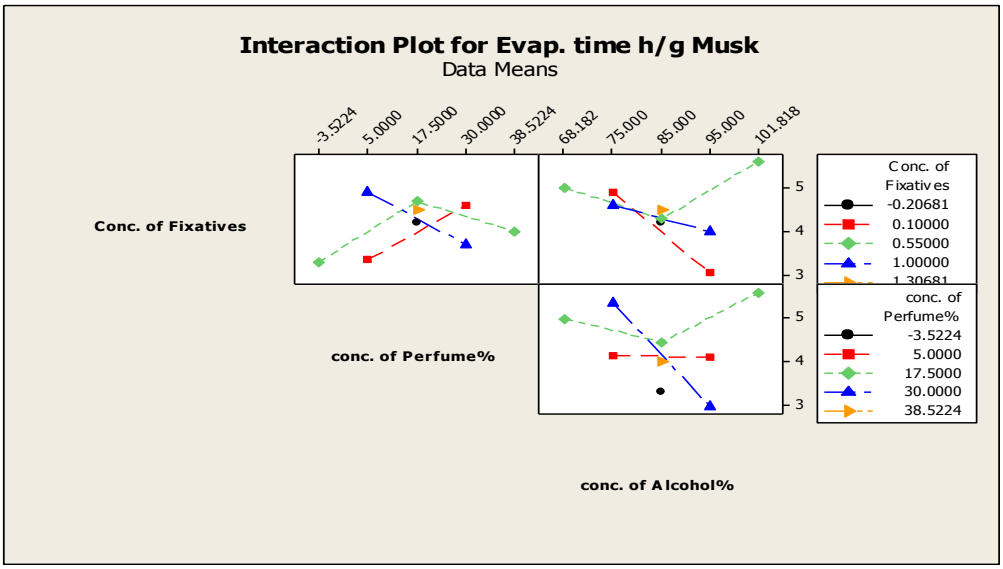


Figure (11) the interaction effect of the evaporation rate with Musk fixative.

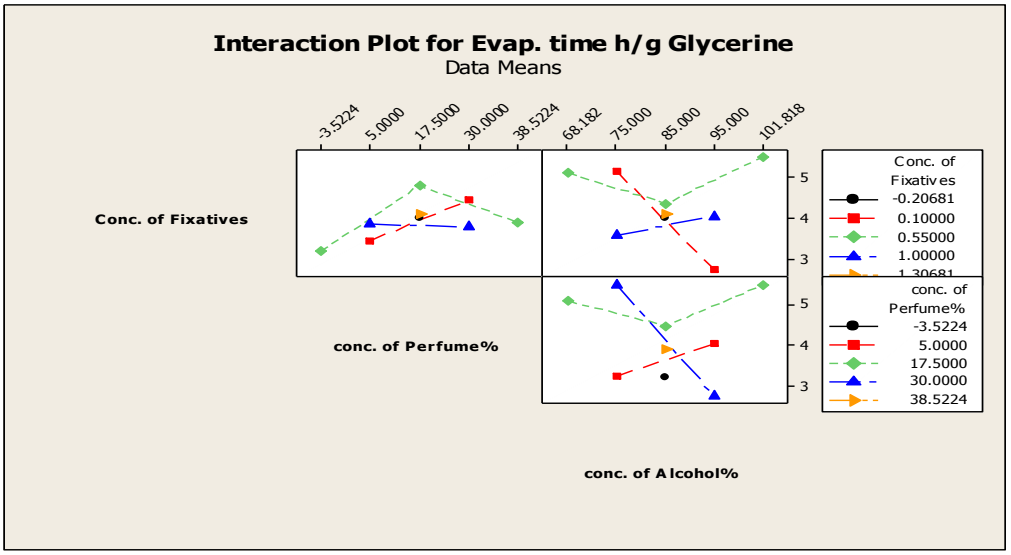


Figure (12) the interaction effect of the evaporation rate with Glycerine fixative.

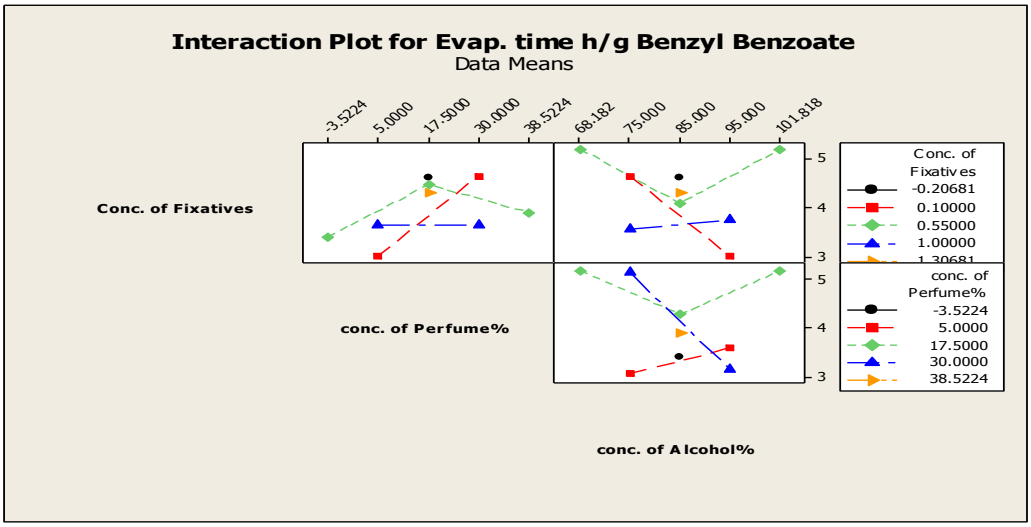


Figure (13) the interaction effect of the evaporation rate with Benzyl benzoate fixative.

CONCLUSIONS

1. The optimum essential oil concentration at maximum time of perfume evaporation (maximum fixation time) depend on the type of fixative material, with Sandal wood fixative materials is 17.5% while with other fixative materials studied the concentrations of the essential oil was 30%.
2. The optimum conditions for perfume formula to get suitable evaporation time (fixation time) for perfume formulation are also depend on the fixative material used it is: 85% Ethyl alcohol, 17.5% perfume and 0.55% fixative when using Sandalwood oil as fixative, while the formula conditions will be (75% Ethyl alcohol, 30% essential oil and 0.1%fixative material of Musk oil, Glycerin or Benzyl benzoate).
3. The regression equations obtained from the four fixative materials studied showed that coefficient of the concentration of the fixative material when it is Sandal have larger values than the coefficients of the concentration of the fixatives when using other fixative materials, which confirm that Sandal wood is more sensitive to concentration added to the perfume on the property of evaporation.

4. The fixation materials used and studied are suitable for perfume formulation and give better results than the brand perfumes studied.
5. Regarding the temperature effect on the rate of evaporation was not studied because all the runs of experiment were executed on the same room temperature during February and March (winter season). So there is no difference on each of the samples prepared and were tested.

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