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Perfume Compositions Containing Natural Resins: Frankincense, Benzoin, and Mastic

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Abstract

The objective of this study was to formulate perfume with natural resins that include frankincense, benzoin, and mastic. In this study, the perfume was formulated using the following ingredients: 2% of each resin; 5% of perfume oil (from Delta aromatic); 4% of glycerin as a humectant; 2% of sodium salicylate as a preservative; and ethanol in distilled water as ingredient's carriers. The fragrant scent is owing to the 11 % of aromatic ingredients in an alcoholic solution. Both benzoin and frankincense are used as scents and fixatives, which can prevent the more volatile fragrance ingredients from evaporating too rapidly. Ten blended experiments of formulated perfume were undergone to evaluate by a sample of people who were selected randomly from Sirte University to choose the best-blended experiment scent among the ten experiments. Therefore, homogeneity, pleasing aroma, strength, and stability were evaluated as quality parameters of this perfumed formulation. However, a number of different approaches for enhanced perfume solubility by using different solvent ratios of ethanol in water have been proposed and applied to deliver the maximum flavors of using resins.

This study will help to reduce chemical content in perfumery manufacturing by using natural sources that have amazing ascent. All the natural resins that are used in this research are purchased from local sources.

Keywords: *Perfumery, frankincense, benzoin, mastic, natural resins, ethanol.*

1. Introduction

The perfumery industry is based on an attractive combination of art and science.[1] Perfume is a complicated blend of certain ingredients in appropriate quantities.[2-3] The fragrant scent is as a result of the 10–15% of aromatic components in an alcohol solution.[2] These ingredients may be natural products of plant or animal origin as well as synthetic materials.[2] The characteristic odour of a single material is called a note.[3] Perfumes is based on three layers of notes, referred to respectively as top notes, middle notes and lower notes.[4] The top note, sometimes called the head note, head or outgoing note, which consists of volatile fragrances to

give a good initial impression.[4] The middle note, sometimes called the heart note, lasts for several hours and is the most prominent within the fragrance.[5] The middle note is usually combinations of spicy, floral, or fruit scents, extracted from cinnamon, clove, fir, cypress, juniper, marjoram, pine, rose, rosemary, thyme, and yarrow.[5] As indicated by their name, the base notes will serve to determine the chief characteristic of the perfume, the sense of which will last hours on end and will be essentially responsible for the success of the perfume, if any.[6] Base notes are products of low volatility and high tenacity.[6]

Perfume is conventional perfuming compositions that have a fragrance profile characterized by the classical fragrance pyramid three-tiered structure, which contains a higher amount of the base notes, a medium amount of the heart notes, and a lower amount of the top notes (see Fig. 1).[7]

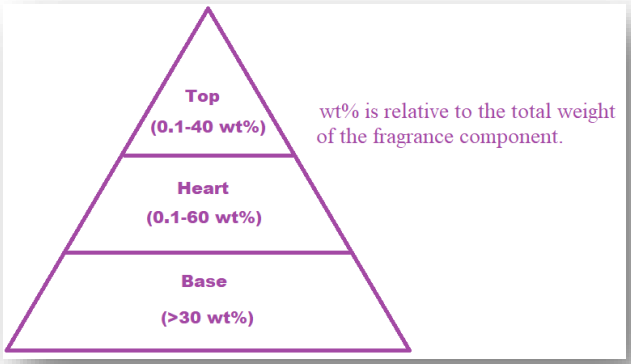


Figure 1: The fragrance pyramid.[8]

The active components of used botanical resins are summarized in Table 1 and Fig. 2.

Table 1: Botanical names and main chemical composition of used resins.

Resin name	Botanical name	Family name[9,10]	Main chemical composition
Frankincense	*Boswellia* spp.	Burseraceae	mono-terpenes (13%) and diterpenes (40%), ethyl acetate (21.4%), octyl acetate (13.4%) and methylanisole (7.6%), lupane **1** and dammarane-type triterpenoids: (3*β*,20(*S*)-dihydroxydammar-24-ene **2,** 20(*S*)-Protopanaxatriol **3**).[11,12]

Mastic	*Pistacia lentiscus*	Anacardiaceae	Monoterpenes (24%): α-pinene **4**, β-myrcene **5**, limonene **6**, camphene **7**, and β-pinene **8**; triterpenoid acids: moronic **9**, oleanonic **10**, masticadienolic **11**, masticadienonic **12**, and isomasticadienonic **13** acids.[11,13,14]
Benzoin	*Styrax benzoin and Styrax tonkinense*	Styraceae	coniferyl cinnamate **14**, cinnamyl cinnamate (styracin) **15**, coniferyl benzoate **16** associated with cinnamic **17,** and benzoic **18** acids. Traces of benzaldehyde **19** and vanillin **20** and styrene **21**.[11]

Moreover, some medical usages of used resins are summarized in Table 2.

Table 2: Some usages of used resins.

Resin Name	Medical uses
Frankincense	Sedative and aromatherapy.[4,10,15]
Mastic	Antitumor, stomach-soothing, and heartburn.[4,10]
Benzoin	Cough and bronchitis.[10]

Natural flavors are prepared by extraction from plants or by enzymatic or microbial processes.[16]

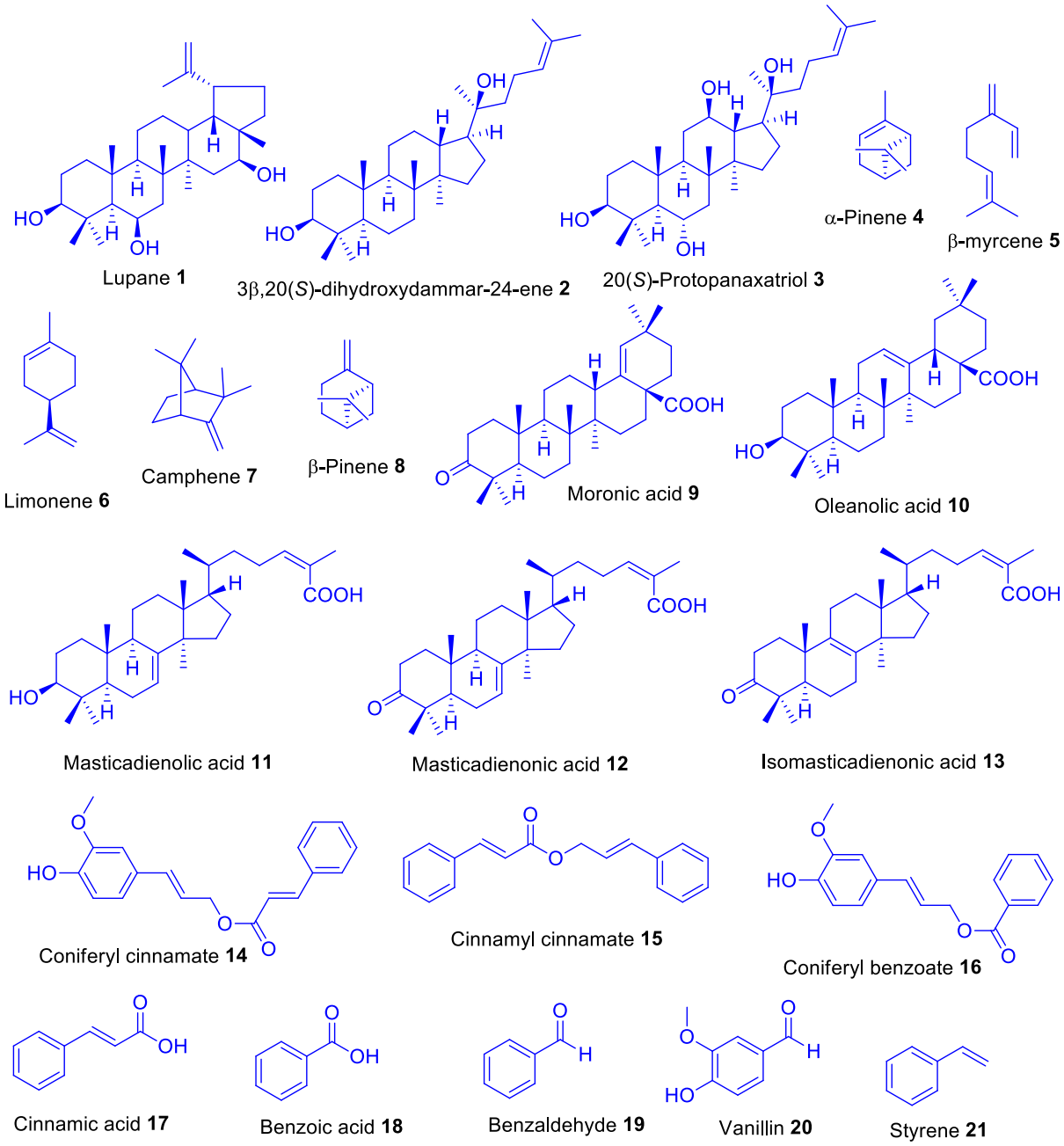


Figure 2: chemical structures of main components of used resins.

2. Materials And Methods

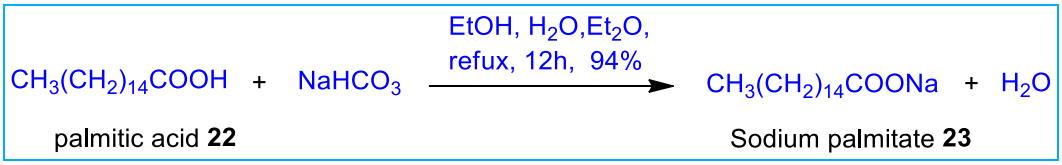
2.1.Chemicals and equipment:

Ethanol was purchased from BDH. Glycerine was bought from Fluka chemika. All used natural resins; frankincense, benzoin, and mastic were purchased from local distributors in Libya and used without further modification. Perfume oil was purchased from Delta aromatics (www.deltaaromatic.org). Samples mixing were carried out using a Vortex mixer (Bio Cote).

PH measurements were performed using a pH Benchtop meter (Orion 2 star, Thermo Scientific).

2.1 Preparation of Sodium Palmitate

Equation 1: Synthesis of sodium palmitate **23**



The following steps are used to prepare sodium palmitate **23**:

i. To a three-necked round flask equipped with thermometer, magnetic stirrer and condenser, the palmitic acid **22** (5 g , 17.9 mmol) and ethanol (50 mL) were added.

ii. The mixture was mixed for 5 minutes and then distilled water (2.8 mL) and sodium bicarbonate (2.2 g, 26.2 mmol) were added to the mixture.

iii. The mixture was heated under refluxing condensation for 12 hours. Then the mixture left to cool to 42°C.

iv. With continuous stirring, the resulting mixture was diluted using diethyl ether (11 mL), and the mixture left to stir for an additional 4 hours. After reducing the temperature to 25 °C, the White precipitated material is filtered by the Buchner funnel and then left to dry.

v. The product was divided into small portions using a spatula and placed under vacuum air at 25 °C for 16 hours, the sodium palmitate **23** is obtained as a white solid (5.1 g, 94%), mp 287-290 °C (283 -290 c ◦ Lit).[17]

2.2 Testing of Solubility of Natural Resins and Surfactant

Two main solvents, water, and ethanol have been used to test the solubility of aromatic resins, which were thoroughly grinded well. It found that the best soluble resin is mastic and the worst solubility rein is benzoin. The solubility of them is summarized in Table 3.

Table 3: Results of the solubility of used natural resins and surfactant.

Solvent Ingredient	Ethanol (cold)	Ethanol (hot)	Water (cold)	Water (hot)
Mastic	Good solubility	Good - solubility	Low-solubility	soluble
Benzoin	Low-solubility	Low-solubility	Low-solubility	Low-solubility
Frankincense (Bitter)	Low-solubility	Low-solubility	Low-solubility	Soluble
Frankincense (sweet)	Low-solubility	Low-solubility	Low-solubility	Soluble
Sodium palmitate	Insoluble	Low-solubility	Insoluble	Soluble

2.3 Preparation of Perfume Formulation

The perfumed formulation consists of the ingredients that are shown in Table 4:

Table 4: The weight percentages of the ingredients of the perfumed formulation with the role of each.

Phase	Ingredient Name	W/W%	Purpose
A	Distilled water	75.0	Solvent
	Glycerine	3.0	Moisturizer
	Mastic	2.0	Polymer resin aromatic
	Frankincense	2.0	Polymer resin aromatic
B	Ethyl alcohol	5.0	Solvent
	Sodium palmitate	4.0	Surface tension reducer
	Fragrance oil	5.0	Oily perfume
	Benzoin	2.0	Polymer resin aromatic
C	Sodium salicylate	2.0	Preservative

To prepare the perfume, the following steps are taken:

i. The contents of the aqueous phase (A) have been weighted and mixed together well.
ii. The contents of oil phase (B) have been weighted and mixed together well.
iii. The contents of the aqueous phase were slowly added to the contents of oil phase and preservative.

iv. The ingredients were stirring together using Vortex Mixer with speed of 100 to 3200 rpm (RPM) to obtain a good homogeneous mixture.

v. The mixture has been left to test the consistency and stability of its fragrance components for several days.

3. RESULTS AND DISCUSSION

3.1 Preparation of Perfume Formulation

Basic ingredients of the perfume formulation were selected based on their outstanding aroma and aromatic stability. Natural aromatic resins, which are represented in benzoin, mastic and frankincense (bitter and sweet), have a beautiful scent. Benzoin is classified as part of Basic Notes because its smell remains for a long time. In addition, it is characterized by a pretty aromatic resin and is available in the local market at a convenient price.

Glycerine **24** is a well-known triglyceride and is one of the most widely used ingredients in perfume formulations. It is a water-soluble, odourless, colourless, and emollient moisturizer that is used to reduce and treat dry skin caused by the use of alcohol in aromatic formula.[18]

Sodium salicylate **25** was selected as a stabilizer and preservative because it has an ionic nature as a hydrophobic and hydrophobic ends. Besides it is authorized for use by the FDA (Food and Drug Administration) in simple proportions in cosmetic formulations as a preservative.[19] It is also used as an anti-steroidal anti-inflammatory medicine.[19]

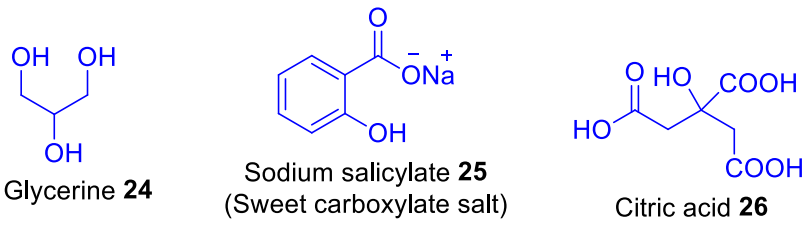


Figure 3: chemical structures of some components of formulas.

Instead of using sodium salicylate, citric acid **26** can be used as a preservative. It is a weak organic acid which exists in citrus and can be used as a natural preservative material and acted also as an antioxidant. [25]

Sodium palmitate was prepared *in vitro* according to the previously described method (Duceppe, J-S., et al) by reacting palmitic acid with sodium bicarbonate and is therefore the

only surfactant used in the this study.[20] Sodium palmitate (Fig. 4) has an ionic nature as a hydrophobic and hydrophobic ends.

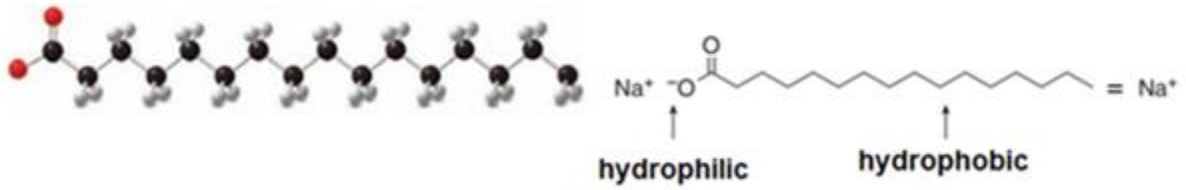


Figure 4: Nature arrangement of user surfactant.

3.2 Study of the weighted ratios of aromatic components in the fragrance composition

The components of the aromatic composition and the role of each of them were mentioned in table 4. These ingredients were tested to obtain the best solubility and extract the beautiful flavor of the resins. The used resins undergo to test their flavor's solubility in water as an alone solvent or as a combination with ethyl alcohol in certain proportions. Many attempts in this study were done to obtain the best extraction of aromatic ingredients and the best liquid texture.

The weight ratios of the aromatic resins used in the composition were 6%, so that each proportion did not exceed 2%. These resins were well ground and the least soluble was benzoin. The oil fragrance was used with a weight ratio of not more than 5%.Oil fragrance used by weight has not exceeded 5%.

The aromatic components of both frankincense and mastic are terpenic resins and therefore can be considered among the top notes and the heart notes except Benzoin, which is among the phenolic resins and it can be considered within the base notes that stay longer. While the oil fragrance is considered one of the top notes. [4,11] The general scheme of our perfume strategy is shown in Fig. 5. The scheme is divided into three specific tasks: (i) top notes (fragrance oil and traces of resins aromas), (ii) heart notes, and (iii) base notes.

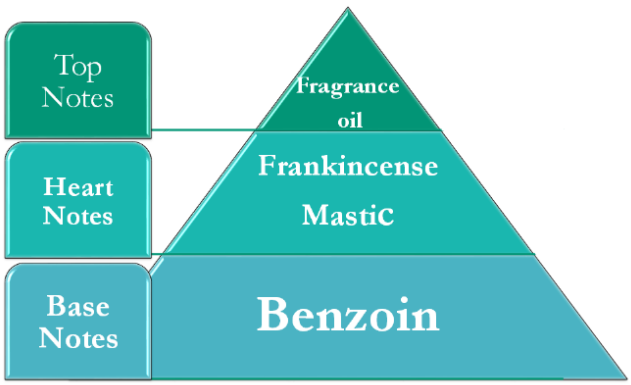


Figure 5: Triangle scheme nearly represents aromatic components of the notes.

To solve each of these tasks, comprehensive understanding of interactions between volatile components and solvent. Otherwise, the ability of solvent to extract these flavours is the most important task.

3.3 Study of Nature Aromatic Composition Free of Alcohol (Sample A)

During the first phase of this study, our efforts involved attempts to isolate the perfume formula with using water as the only solvent. Water is considered the safest solvent for health and for using on the skin, so an aromatic formula is prepared using the water as only solvent.

The nature of the surfactant (sodium palmitate) tends to help solvation of both aqueous and oily phases because it has a hydrophobic end and a hydrophilic end that makes the texture to form an emulsion with both water and oil phases. The result obtained after mixing the aqueous and oily phases with the presence of a surfactant was a heavy-textured emulsion (soapy) with a beautiful aromatic scent.

The fragrant components of the alcohol-free sample are shown in Table 5, so that the water ratio was 80% and there was no bacterial or fungal contamination occurred to the sample, although it remained for over six months at room temperature. This is could be attributed to use of sodium salicylate as preservative.

Table 5: Composition of aromatic alcohol-free sample A.

Phase	Ingredient Name	W/W%
A	Distilled water	80.0
	Glycerin	3.0
	Mastic	2.0
	Frankincense	2.0
B	Sodium palmitate	4.0
	Fragrance oil	5.0
	Benzoin	2.0
C	Sodium salicylate	2.0

3.4 Studying the Impact of Used Solvents on the Solubility of Aromatic Ingredients to Obtain the Best Extraction [Samples from (A) to (N)].

The solubility of the aromatic components was tested in different proportions of ethyl alcohol in water (Table 6). It has been observed that the presence of ethyl alcohol increased the solubility of the aromatic ingredients, and make the texture more clear. Volumetric ratios of alcohol in water were gradually increased by 5% for samples B to N, and any change affecting the smell and emulsion texture was observed.

Table 6: Effecting of using of different volumetric ratios of alcohol in water on texture and solubility of perfumed samples.

Formula symbol	%W/W of water	%W/W of ethanol	Perfume texture
A	80.0	0.0	Soapy
B	75.0	5.0	Soapy
C	70.0	10.0	Soapy
D	65.0	15.0	Soapy
E	60.0	20.0	gel
F	55.0	25.0	Emulsion
G	50.0	30.0	Emulsion
H	45.0	35.0	Emulsion

I	40.0	40.0	Emulsion
J	35.0	45.0	Liquid
K	30.0	50.0	Liquid
L	25.0	55.0	Liquid
M	20.0	60.0	Liquid
N	15.0	65.0	Liquid

Furthermore, Study of the texture of the aromatic formulations without the surfactant is tested in different considered samples (Table 7). It is possible to exclude surfactants for perfumed samples that contain over 15% percent of alcohol as a solvent. Usually, the surfactants used in alcohol-free perfumes or alcohol content not exceeding 15%. As a result of that exclusion, it was noted that the transparency of the aromatic solution increased to clear solution in an aromatic formula containing 39% of alcohol (sample O), while the sample texture became an emulsion with a reduced ratio of 40% alcohol (sample I).

Table 7: Effecting of using different volumetric ratios of alcohol in water on perfume texture.

Formulation symbol	%W/W of water	%W/W of ethanol	Perfume texture
O	45.0	39.0	Liquid
P	40.0	44.0	Liquid
Q	35.0	49.0	Liquid
R	30.0	54.0	Liquid
S	25.0	59.0	Liquid
T	20.0	64.0	Liquid

3.5 Calculations of pH for Formulations Samples

The pH calculated values of all samples were in the range from 6.47 to 6.67, due to the fact that the components are not acidic or basic. The surfactant is a salt of salicylic acid has not cause acidity of the pH values of the aromatic composition. Overall, the results of pH reading of all samples showed that it is in the neutral range.

3.6 Evaluation of Aromatic Sampling Scents and Selecting The best Combination between the Prepared Samples

According to the ephemeral sense of smell of any fragrance, it found that all formulas have a good aroma and high aromatic stability. All formulas were remained after preparation for more than six months preserved at room temperature and still retain their stability and scents.

Seven people were randomly selected from the University of Sirte to test the best prepared aromatic formulation. The marks were given from every seven people (each person was denoted by the symbol P as an abbreviation of the word person) for each formula, and the mark given is of the highest score 10. The given marks are shown in Table 8.

The assessment of the perfume blend was then calculated from Equation (2).[21]

Table 8: Marks obtained for each sample submitted for evaluation by seven people.

Person code	K	L	M	N	O	P	Q	R	S	T
P_1	8	8	9	7	8	7	5	8	7	5
P_2	7	6	8	9	8	7	6	8	7	8
P_3	10	10	10	10	10	10	10	10	10	10
P_4	10	10	10	10	10	10	10	10	10	10
P_5	8	9	8	8	10	7	9	9	8	7
P_6	8	6	6	6	9	9	8	8	7	8
P_7	8	9	7	7	10	10	10	10	8	8

Equation 2: calculation of perfume efficacy

$$Evaluation\ of\ Pefume\ formulation\ (EPF) = \frac{total\ marks\ obtained}{number\ of\ people}$$

$$EPF\ for\ sample\ O = \frac{65}{7} = 9.2$$

The composition is the one that got the highest mark (9.29 out of 10) was considered the best, which is for sample O.

Table 9: Showing average values (rating score) for tested samples.

Sample	Evaluating marks	Sample	Evaluating marks
K	8.42	**P**	8.57
L	8.28	**Q**	7.71
M	8.28	**R**	9.00
N	8.71	**S**	8.14
O	9.29	**T**	8.00

3.7 Comparison of the Ratios of Solvents Used to Extract Aromatic Composition with the Degree of Evaluation

By comparing the percentage of solvents used to extract the aromatic formula with the evaluation score obtained, it found that the best aroma of the sample contains 39% alcohol in water (sample O), while the lowest rating for the sample containing 49% alcohol in water (sample Q). The rest of the samples received a good rating and converged score from 8.00 to 9.00. On other hand, it is observed that any increase in alcohol over the 39% did not contribute to improve the aroma. Samples O to T do not contain any surfactant because their alcohol ratios are relatively high.

Table 10: Explanation of the ratios of solvents used in the extraction of aromatic composition and the degree of evaluation.

Formulation symbol	%W/W of water	%W/W of ethanol	Evaluation
K	30.0	50.0	8.42
L	25.0	55.0	8.28
M	20.0	60.0	8.28
N	15.0	65.0	8.71
O	45.0	39.0	9.28
P	40.0	44.0	8.57
Q	35.0	49.0	7.71
R	30.0	54.0	9.00

S	25.0	59.0	8.14
T	20.0	64.0	8.00

The fragrant composition components of the 10 samples are arranged downward by rating score so that:

O > R > N > P > K > L = M > S > T > Q

4. Conclusions

The fragrance ratios used in the aromatic composition are only 11% as shown in Fig. 6. So the perfume can be classified as a type of Eau de perfume.

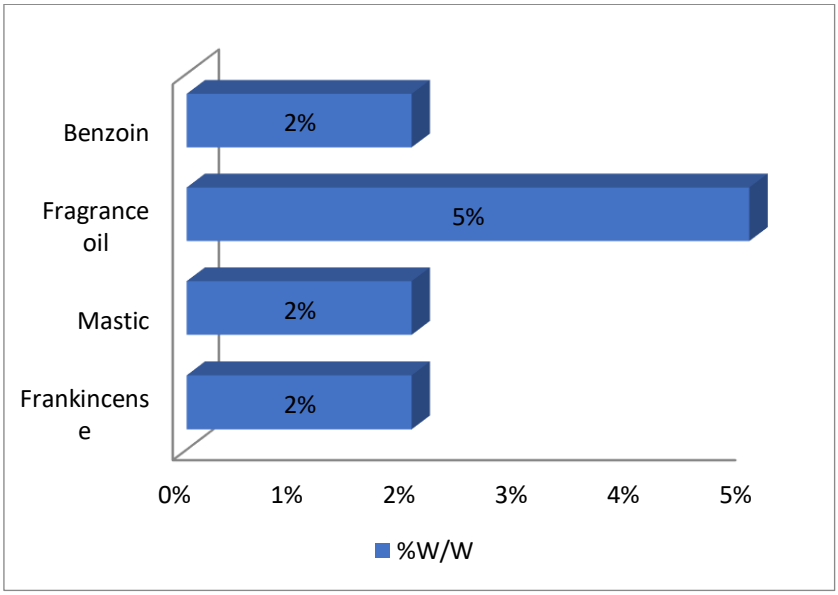


Figure 6: Ratio of aromatic ingredients in aromatic formulation.

Thus the approved aromatic composition of the best given aroma is shown in table 8, noting that the same aromatic composition differs only in solvents carrying the composition components.

Table 11: Components of the aromatic composition of the best sample in terms of odour and strength.

Phase	Ingredient Name	W/W%

A	Distilled water	45.0
	Glycerine	3.0
	Mastic	2.0
	Frankincense	2.0
B	Ethyl alcohol	39.0
	Fragrance oil	5.0
	Benzoin	2.0
C	Sodium salicylate	2.0

5. Recommendations

It is recommended that further research be undertaken in the area of perfumes from natural ingredients, thereby reducing the chemical components manufactured in these products. Also continue to look into the use of surfactants commensurate with perfumes based on the reduction of alcohol as a solvent.

6. Acknowledgments

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