



Original Article

## Formulation of Essential Oil-based Air Freshener Gel

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**Abstract:** Meanwhile, the health risks caused by chemical or volatile organic compounds from gel air fresheners, and odorremoval products have been reported. However, these products are continuously present in daily life. The essential oil-based air freshener gels are safe and available price products for the consumer and for human health protection. They consist of natural gelling agents (carrageenan, xanthan gum), natural fragrances (essential oils and aroma extracts), a surfactant (polysorbate 20), co-solvent (ethylene glycol), sodium benzoate, sodium chloride, and distilled water at varying concentrations. The essential oils extracted from pomelo peels and aerial parts of lemongrass and peppermint act as flavoring and antimicrobial agents. Scent formulas of D-JA, D-PO, and D-FV blended from natural fragrances are conducted on the various gel formulas (I, II, and III). The most significant product is formula I (D-PO), recorded at 0.0121 of specific gravity, a syneresis rate of 18.91%, and pH 4.88. This formula meets the given criteria and panelists. Additionally, the air freshener gels containing essential oils as bioactive compounds present antibacterial activity against *Escherichia coli* and *Staphylococcus aureus*., especially the formula with lemongrass essential oils and blended essential oils. The essential oils on air freshener gel can inhibit the growth of bacteria and are safe for the consumer.

**Keywords:** Air freshener gel, essential oil, extract, carrageenan, natural fragrance, antibacterial activity.

### 1. Introduction

Fragrances are considered important sensory attributes in the formulation of fragranced products such as fine fragrances or functional perfumes like soaps, household cleaners, and detergents [1]. In air freshener

products, fragrance plays a significant role in spreading scent for relaxation and cleaning the air. Carrageenan gels use carrageenan as a natural gelling agent. It is a viscous material contained in the red-seaweed group. These gels are divided into hydrophilic gels [2].

Air fresheners and consumer products, typically emitting fragrances, are used in homes or commercial interiors. Some air fresheners contain chemicals that provoke allergy and

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asthma symptoms or are toxic. The first modern air freshener appeared in 1948. The product, using technology developed by the military to dispense insecticides, was a pressurized spray containing about 1% perfume, 24% alcohol or other solvents, and 75% chlorofluorocarbon (CFC) propellant. It can deliver a fine mist of fragrance, which remains suspended in the air for a long period [1, 2].

Air freshener scent gels using natural safe ingredients for the environment have been studied recently. Semoff and Sarraf (1997) presented the method of making a gel air freshener comprising an aqueous gel, a fragrance, a surfactant, and a co-solvent [3]. Also, the air freshener in the gel dosage form is simpler in storage and packaging [4]. Triplett et al., had the patent recorded for the invention of gel air freshener and the method of manufacturing same. In this work, the gel was improved in composition for delivering volatile materials such as fragrance oils, fragrance solutions, deodorants, and insecticides [5]. Formulation and methods of preparing gel air fresheners, including transparent gel air fresheners containing water, a gelling agent, a cross-linking agent, a co-solvent, and fragrance oil, are well known in the prior art. A cross-linking agent to the gelling agent and other gel components is introduced to achieve the desired gel properties, such as optimum clarity and gel structure [6]. Recently, many studies on air fresheners have been still reported. Hutagaol reported the formulation of air freshener gel with carrageenan as a gelling agent, lemon oil as a fragrance, and patchouli oil as a binder [7]. Amra Bratovčić used fragrance compounds found since antiquity to freshen the air or mask the odors [8]. Eden et al. examined the active component of *Cymbopogon winterianus* (*Java citronella*) oil as a green mosquito repellent [9].

Essential oil, a natural fragrance playing an important role in air freshener products, cannot be absent. With the increasing demand for natural products, health safety, and long shelf

lives without affecting their quality, the presence of essential oils acting bioactive as an alternative to chemicals is the best choice for the preparation of the scent freshener. By using the Vietnamese's favorite essential oils extracted from plant species cultivated commonly in Vietnam, the scented air freshener gel product was studied and made with a fragrance that both smells pleasant and safe for the customer.

## 2. Experimental

### 2.1. Materials

Fresh plant materials of pomelo (peels), lemongrass (aerial parts), and clove (buds) were collected in the Mekong Delta area (Vietnam) from March to June 2020. The fresh peppermint materials were collected from a home garden (Thu Duc district, Hochiminh City, Vietnam) in July 2020. Vanilla essential oils, *Styrax benzoin* resin, carrageenan, xanthan gum (refined, E415), ethylene glycol, sodium chloride (NaCl), sodium benzoate (E211), and polysorbate 20 (Tween 20) were purchased from Fabulous Frannie (USA), Bio Source Naturals (USA), The World's Finest (Phillippines), Kelco (China), GH Tech (China), Xilong Scientific (China), Eastman (USA), and Bioreagents (USA), respectively. Distilled water was obtained from the organic laboratory.

### 2.2. Methods

#### 2.2.1. Extraction of Essential Oils

The essential oil was distilled by steam distillation or hydrodistillation method depending on the material nature described by Anton de Groot (2016) [10]. Each 15 kg of fresh material was suspended in a distillation system equipped with a 50 L still and worked out for 1.5-2.0 hours. The essential oil was separated from distillate water by centrifugation and funnel extraction. The oil product was dried with  $\text{Na}_2\text{SO}_4$  anhydrous, stored at a cool place, and kept from the sunlight.

### 2.2.2. Building the Scent Pyramid of the Fragrance

The fragrance (Figure 1) was made up of 0-30% top notes, 30-70% middle notes, and 30% base notes. Blending ingredients of the top note (pomelo essential oils), middle note (lemongrass essential oils, peppermint essential oils, clove essential oils), and base notes (vanilla extract, styrax benzoin resin extract) are according to the proportion built by the scent pyramid shown in Table 1.

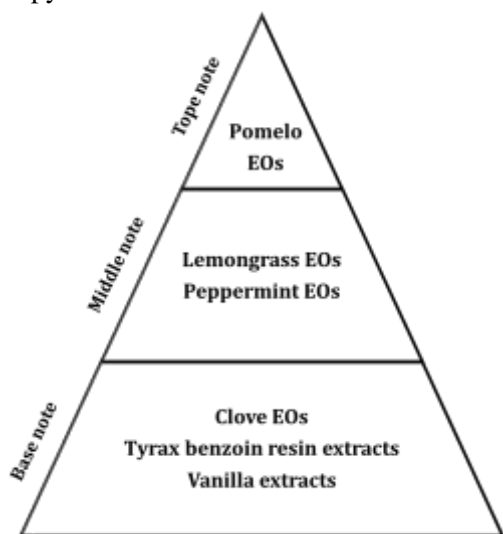


Figure 1. Scent pyramid of fragrance.

Table 1. Ingredients of scent formulas presenting fragrances used in air freshener gel

Fragrant ingredients	Volume (ml)		
	D-JA	D-PO	D-FV
Pomelo essential oil	0	70	25
Lemongrass essential oil	70	0	30
Peppermint essential oil	0	0	5
Clove essential oil	0	0	10
Vanilla extract	10	10	10
Styrax benzoin resin extract	20	20	20

### 2.2.3. Formulation of Air Freshener Gel

Three scent formulas were composed of carrageenan, xanthan gum, sodium benzoate, ethylene glycol, polysorbate 20, NaCl, distilled water, and fragrance. Table 2 shows the formula of the air freshener gel.

Table 2. Formulas of air freshener gel

Ingredients	% of weigh		
	I	II	III
Formula			
Carrageenan	3.0	2.0	2.5
Xanthan gum	0.5	2.0	1.0
Sodium benzoate	0.30	1.0	1.0
Ethylene glycol	8.2	5.0	6.0
Polysorbate 20	-	-	0.5
NaCl	1.0	1.0	1.0
Distilled water	83	85	84
Fragrance (ml)	4.0	4.0	4.0

### 2.2.4. Preparation of Air Freshener Gel

Table 2 present the amount of each component in the gel samples. First, 1.0 g of NaCl and the exact amounts of carrageenan and sodium benzoate were introduced to 50 mL of distilled water under stirring, followed by heating at 75 °C and 65 °C. A definite amount of xanthan gum and 50 ml of distilled water were slowly added to the solution during heating. Ethylene glycol and polysorbate 20 were dissolved into the mixture. The fragrance (4 mL) was homogenized into the mixture after adding distilled water to obtain 100 g of product. The mixed solution was stirred until homogeneity, eventually inserted into the mold, and stood at room temperature.

### 2.2.5. Product Quality Evaluation/Assessment

The quality control tests include:

- i) Analysis of chemical composition: fragrance;
- ii) Ocular assessment: organoleptic property;
- iii) Physical controls: specific gravity, pH;
- iv) Microbial growth test: antibacterial activity;
- v) Happiness function: scent (syneresis rate), Hedonic test.

### 2.2.5.1. Analysis of the Chemical Composition of Fragrances

The volatile compounds of fragrances were analyzed on an Agilent 7890A GC equipped with an HP-5MS capillary column (30.0 m x 0.25 mm x 0.5  $\mu$ m) coupled to an Agilent Mass Selective Detector 5975C VL MSD Triple-Axis. Helium was the carrier gas at 0.9 mL/min. The constant pressure mode was at 13.209 psi on the GC program. The injection temperature was 250  $^{\circ}$ C; the injection volume was 1.0  $\mu$ L; the splitless mode and the ionization voltage were 70 eV. The rate of the column temperature was 3  $^{\circ}$ C/min to 240  $^{\circ}$ C. The components of the oils were identified by gas chromatography by comparison of their linear retention indices (programmed retention indices) calculated relative to the homologous series of C8-C22 *n*-alkanes using the equation propounded by van den Dool and Kratz in 1963 [11, 12]. The constituents were identified by comparing their mass spectra with those of standard compounds registered in the NIST 2017, and the Wiley 8<sup>th</sup> edition libraries [13]. Moreover, the results were confirmed by collating the calculated retention indices of each constituent with those reported in the compilation of retention indices published by Adams [14]. The relative percentage of each constituent in the oil was determined by peak area normalization. No response factors were calculated.

### 2.2.5.2. Ocular Assessment

The organoleptic property of the gel sample was assessed by recording color and odor.

### 2.2.5.3. Physical Controls

The physical properties of gel samples were controlled by determining specific gravity and pH meter.

The specific gravity of the gel (Equation 1) was evaluated by the ratio between the density of the gel (Equation 2) and the density of the water, which is given that the reference substance is at room temperature.

$$\text{Specific gravity} = \frac{\text{Density of gel}}{\text{Density of water}} \quad (1)$$

In which:

$$\text{Density of gel} = \frac{\text{Mass of gel (g)}}{\text{Volume of gel (mL)}} \quad (2)$$

The pH was determined using a Mettler Toledo Sevencompact pH meter. The formulations were prepared by dissolving 1 g of gel in 10 mL of distilled water, which was further tested with a calibrated pH meter at room temperature.

### 2.2.5.4. Microbial Growth Test

The evaluation of antibacterial activity was carried out on three fragrance samples prepared. The Kirby-Bauer antibiotic tests (also referred to as the disc diffusion antibiotic sensitivity tests) were performed according to the National Committee for Clinical Laboratory Standards (NCCLS, 2001) [15, 16]. The reference strains *Escherichia coli* ATCC 85922 and *Staphylococcus aureus* ATCC 25023 were used in this research.

### 2.2.6. Happiness Function

Syneresis was evaluated by the expulsion of liquid from a gel sample at room temperature. The gel sample's weight loss was recorded weekly for 2 months. The percentage of total liquid evaporation was calculated following Equation 3.

$$\text{Total evaporation (\%)} = \frac{M_n - M_{n-1}}{M_o} \times 100 \quad (3)$$

Note:

$M_n$ : the weight of gel at the time of weighing;

$M_{n-1}$ : the weight of gel in the previous week;

$M_o$ : the weight of gel at the initial time.

The Hedonic test was carried out according to ISO 11136:2014 (TCVN 12751:2019). The measurement of Hedonic responses was adapted category-ratio scaling by LAM (labeled affective magnitude) scale and the 5-point hedonic scale on various of time intervals, fragrances, criteria intervals, and formulas [17, 18]. The questionnaire relating the scent/aroma of fragrance, texture, acceptability, and spreading quality of air freshener gel products made from various formulas was provided to the panelists.

### 3. Results and Discussion

The process of study was carried out following the workflow shown in Figure 2.

#### 3.1. Analysis of the Chemical Composition of Fragrances

The result of GC/MS analysis (Table 3) showed that the dominant components of fragrance were dependent on the characteristic feature of the contributing ratio of essential oils.

The fragrance of D-JA and D-PO was dominated by 85.65% of citrals and 78.40% of limonene, respectively. The D-FV fragrance

was presented with various constituents such as limonene, citrals, eugenol, and linalool.

These results indicated that the combination of volatile essential oils (lemongrass, pomelo, clove), styrax benzoin resin extract, and vanillin extract in fragrances could be potentially bioactive materials for air freshener products.

The application of fragrance built on three formulas of base gel gave nine air freshener gel products. To obtain the fine product, evaluation and assessment of product quality were carried out.

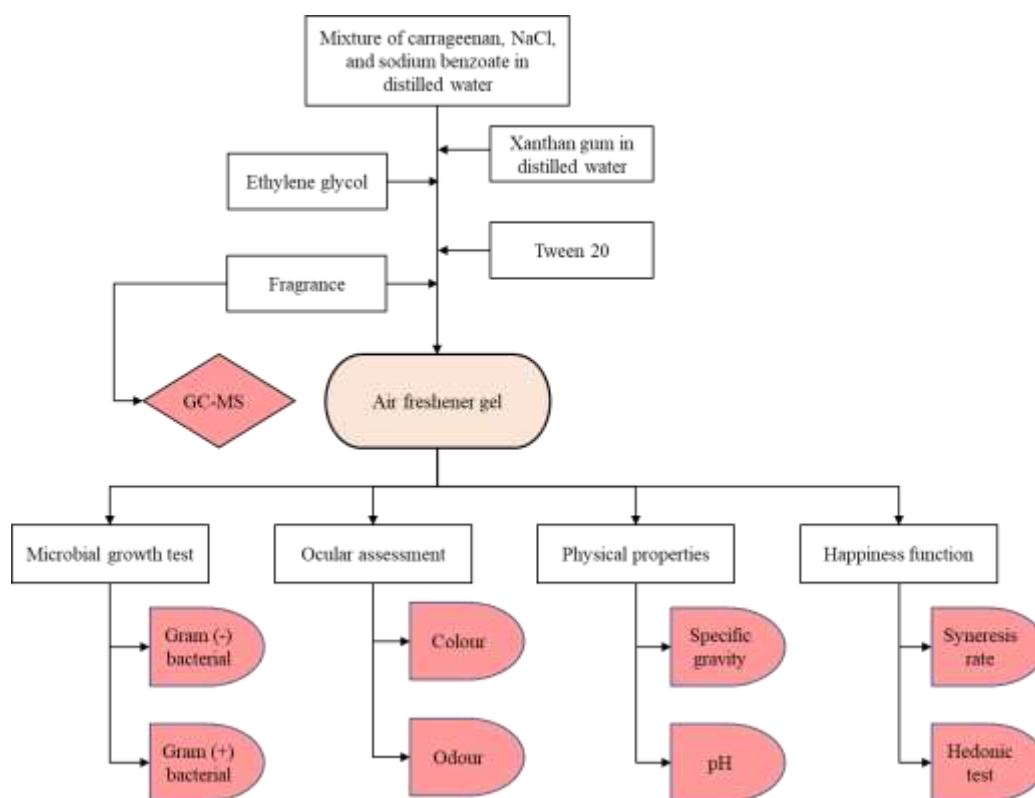


Figure 2. Workflow of study.

Table 3. Main constituents of the three fragrance samples

No.	Main compound name	LRI		Percentage (%)		
		Obs.	Lit.	D-JA	D-PO	D-FV
1	$\alpha$ -Pinene	974	974		2.30	
2	$\beta$ -Myrcene	988	988		1.10	
3	$\alpha$ -Phellandrene	1002	1002		0.93	

4	<i>o</i> -Cymene	1021	1022		5.65	1.78
5	Limonene	1025	1024	4.34	78.40	30.77
6	$\gamma$ -Terpinene	1056	1054		7.41	3.52
7	Linalool	1096	1095			4.91
8	Terpinen-4-ol	1178	1174	1.25		1.04
9	Neral (citral b)	1235	1235	40.85		11.21
10	Geraniol	1248	1249	1.26		
11	Geranial (citral a)	1265	1264	44.80		12.03
12	Eugenol	1357	1356			12.06
13	Vanillin	1394	1393	0.22		
14	Caryophyllene	1418	1417	1.19		3.74

LRI (linear retention index) on HP-5MS [Obs.: observed, Lit.: literature reported by Adams [14]].

### 3.2. Ocular Assessment

Nine air freshener gel samples (Figure 3) were prepared by the gelling (xanthan gum, carrageenan) and active agents (D-JA, D-PO, D-FV fragrances) according to the formulas in Table 2. Table 4 shows the ocular assessment by 30 panelists.



Figure 3. Air freshener samples.

Table 4. Organoleptic properties of air freshener gel products formulated

Formulas	Fragrances	Organoleptic property	
		Colour	Odour
I	D-JA	Pale yellow	Fresh, grassy
	D-PO	Colourless	Fresh, sweet
	D-FV	Pale yellow	Strong, warm, spicy
II	D-JA	Yellow	Fresh, grassy
	D-PO	Pale yellow	Fresh, sweet
	D-FV	Yellow	Strong, warm, spicy
III	D-JA	White	Fresh, grassy
	D-PO	White	Fresh, sweet
	D-FV	Pale yellow	Strong, warm, spicy

### 3.3. Physical Controls

The physical properties of air freshener products were evaluated on specific gravity and pH, as given in Table 5.

The organoleptic test by 30 panelists indicated that formula I was highly acceptable. Its D-FV fragrance had the longest storage time. Formula III with the long release and storage life of air freshener gel was accepted by the panelists. This formula met the requirements,

namely at a temperature of 35 °C, and can last up to 10 weeks. Overall, these results showed that the storage lifetime was relatively higher than the study of Eden et al. [9], which had the air

freshener gel’s best storage lifetime of approximately 22.40 days. The pH significant value increase from the 10th week gave prominent instability in every formulation.

Table 5. Physical properties of air freshener gel products formulated

Formula	Fragrance	Specific gravity (25 °C)	pH				
			2 <sup>nd</sup> week	4 <sup>th</sup> week	6 <sup>th</sup> week	8 <sup>th</sup> week	10 <sup>th</sup> week
I	D-JA	0.0109	4.65	4.71	4.88	4.92	5.00
	D-PO	0.0121	4.65	4.78	4.81	4.85	4.88
	D-FV	0.0098	4.89	4.91	5.00	5.06	5.12
II	D-JA	0.0135	5.4	5.44	5.56	5.65	5.68
	D-PO	0.0138	5.01	5.12	5.2	5.25	5.17
	D-FV	0.0121	5.18	5.25	5.33	5.48	5.48
III	D-JA	0.0106	5.32	5.36	5.4	5.42	5.4
	D-PO	0.0115	5.18	5.26	5.35	5.42	5.55
	D-FV	0.0120	4.89	4.95	5.08	5.13	5.15

3.4. Microbial Growth Test

The antibacterial activity of fragrance samples made up of the scent of air freshener gel was studied against two strains of pathogenic bacteria: *Escherichia coli* ATCC

85922 and *Staphylococcus aureus* ATCC 25023. The antibacterial activity was determined by the ability to inhibit the growth of bacteria, expressed by the antibacterial diameter inhibition zone (Table 6 and Figure 4).

Table 6. Diameters of the inhibition zones of fragrance samples

Bacterial strain	Diameter of the inhibition zone (mm)		
	D-JA	D-PO	D-FV
<i>Escherichia coli</i> ATCC 85922	Non-bacteria grows	2.13 ± 0.52	8.71 ± 0.34
<i>Staphylococcus aureus</i> ATCC 25023	Non-bacteria grows	6.73 ± 0.71	11.22 ± 0.82

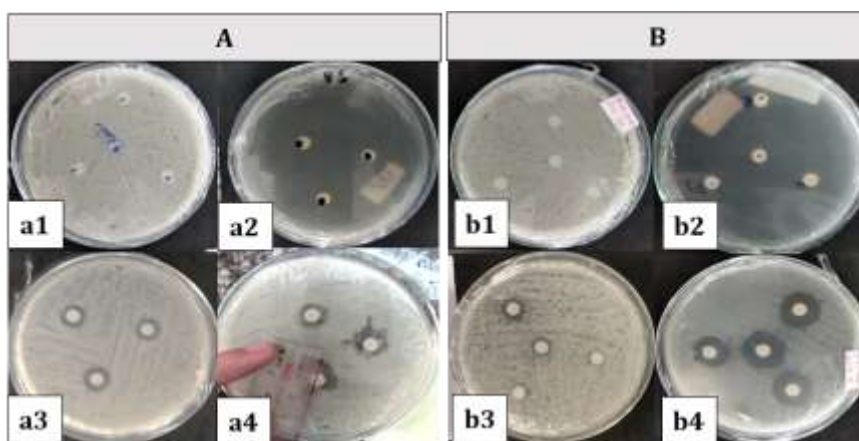


Figure 4. A. Growth of *E. coli* affected by fragrance samples: a1) Control, a2) D-JA, a3) D-PO, and a4) D-FV; B. Growth of *S. aureus* affected by fragrance samples: b1) Control, b2) D-JA, b3) D-PO, and b4) D-FV.

The effects of essential oils on *S. aureus* were stronger on *E. coli*. The inhibition zone of Gram-positive bacteria was larger than Gram-negative bacteria (Figure 4 A, 4 B). Lemongrass essential oil gel had the best bacteria inhibitory activity, up to the 5-flavor mixed gel, while the pomelo oil gel had the lowest antibacterial activity.

### 3.5. Happiness Function

The degree of satisfaction with the scent of air freshener gel from the customer was defined by a

Happiness function. In this work, it was conducted by syneresis rate and Hedonic test.

#### 3.5.1. Syneresis Rate

The actual scent of gel was determined to be the most heavily weighted parameter of the fragrance. The syneresis rate was recorded through the evaporation of the gel product over time. 100 g of prepared gel was observed for weight loss every week at room temperature for two months. The result was shown in Table 7.

Table 7. The weight loss of air freshener gel studied for 10 weeks

Week	Weight (g)								
	I			II			III		
	D-JA	D-PO	D-FV	D-JA	D-PO	D-FV	D-JA	D-PO	D-FV
1	93.76	98.06	98.11	82.13	84.12	95.88	87.68	88.34	84.76
2	91.47	93.76	95.46	78.75	82.10	92.06	85.15	86.51	81.55
3	88.33	90.76	91.65	74.92	79.76	89.78	82.46	84.48	78.78
4	86.69	88.44	89.06	72.99	76.19	86.15	80.09	81.86	75.89
5	83.72	85.69	87.03	70.26	73.97	84.32	78.76	80.15	73.12
6	80.46	83.45	84.75	67.55	72.64	83.22	76.11	78.86	72.88
7	77.32	82.19	82.06	66.08	70.65	82.18	75.02	76.54	71.64
8	75.12	81.22	80.79	64.99	69.53	80.67	73.81	75.15	71.00
9	74.33	80.78	78.98	63.45	68.79	80.06	72.62	74.87	70.12
10	72.91	79.52	77.15	61.99	67.52	79.12	71.89	74.00	68.84
Total evaporation (%)	22.24	18.91	21.36	24.52	19.73	17.48	18.01	16.23	18.78

As shown in Table 7, almost the gel formulated by D-JA fragrance gave high evaporation. The D-PO fragrance gave the evaporation of the gel at the lowest rate. The gel D-FV evaporated slowest in formula II and at the rate between D-JA and D-PO in formula I and formula III. Both D-JA and D-PO fragrances contained 70% of lemongrass and

pomelo, respectively. These essential oils were volatile and gave high evaporation in general. However, lemongrass oil dominated by over 80% of citrals in the D-JA was more polar than pomelo oil dominated by over 90% of monoterpene hydrocarbons (limonene,  $\gamma$ -terpinene, and *o*-cymene) in the D-PO. The D-FV contained both polar essential oils and



non-polar essential oils. The result showed that the syneresis rate of gel products depended mainly on gel formulation and the capacity of the gel base in trapping/linking with/spreading as well as homologating with the aroma ingredients. In this study, the combination of gel bases in formula III helped reduce the evaporation rate of aroma compounds.

### 3.5.2. Hedonic Test

The data obtained from the assessment sheet determined the preference value for each preparation by finding the average yield of all the panelists at the 95% confidence level. The overall results of the Hedonic test at various criteria intervals and formulas were showed in Figure 5. The ones at different time intervals and fragrances named D-JA, D-PO, and D-FV were presented in Figure 6.

Hedonic test results (Figure 5) showed that formula III gave a fine-scented air freshener gel containing 2.5% of carrageenan because its low syneresis rate. Formula II with a low concentration of carrageenan is less preferred. Higher the concentration of carrageenan more ability to maintain scented gel formulation to be better and more favored (formula I). In contrast, the gel with the highest xanthan gum was moderately disliked because of the inhomogeneous texture (formula II). The scent was trapped well by carrageenan. In this way, the scent of the product was prolonged.

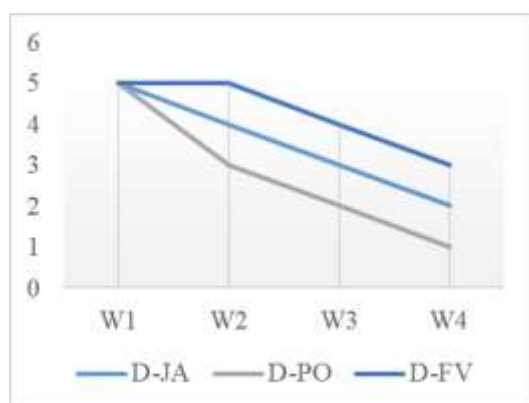


Figure 5. Hedonic test at various time intervals and various fragrance.

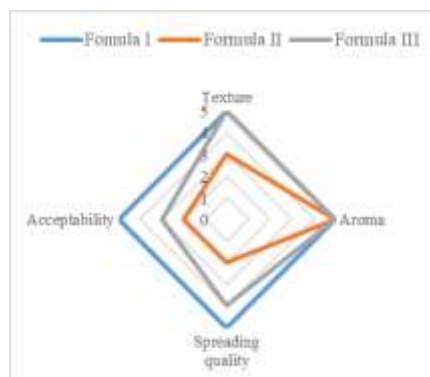


Figure 6. Hedonic test at various criteria intervals and various formulas.

Overall, although having high evaporation, the formula I was appreciated highest by panelists and met the given criteria. Formula II gave the product that the appearance of the surface was non-smooth. Formula III brought the product with the best scent retention but the less fine feeling of spreading quality than formula I did. Among the fragrant formulas, the scent of the D-PO formula was most trapped by the base of gels.

## 4. Conclusion

The air freshener made from formulas built mainly on natural ingredients was stable at high temperatures for up to 10 weeks and showed antimicrobial effects. Using carrageenan as a gelling agent and essential oils and extracts as fragrances have brought promising air freshener gel products, which are health safe, have long shelf lives, have high antimicrobial activities, and are available priced for the consumer. Since lemongrass, pomelo, and peppermint, herbal materials are common and easy to find in Vietnam. In addition, the final prices of the product can be available compared to other current commercial products.

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