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Determination of Iodine and Saponification Values of "Silver Bird" Eucalyptol

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Abstract: *The constant increase in the industrial usage of eucalyptol (cineole) as an additive in pharmaceutical, cosmetic, and confectionary industries, and the tendencies of the greater population to retire from active chemotherapeutics to herbal and natural practices, made this research an interesting if not compulsory exercise so as to ensure safety and industrial feasibility. Sample of "Silver Bird" brand eucalyptol, the most commonly available brand in the country, was obtained from a local pharmaceutical chemist. It was then utilized in the determination of iodine and saponification values, which were found to be 17.956 and 15.848mg KOH/g respectively. The two chemical parameters determined are very essential in understanding the potentials of an oil or fat, especially in the soap making industry.*
Keywords: *Eucalyptol, Iodine Value, Saponification Value, Silver Bird.*

I. Introduction.

There is an increase in the patronage of natural medicinal and cosmetic products of which eucalyptus oil (eucalyptol) is not an exception. Individuals of different caliber tend to prefer traditional (herbal) products, rather than chemical-based products that are always available in the market. Eucalyptol, being one of the natural essential oils, is daily gaining grounds in the pharmaceutical and cosmetic industries.

Eucalyptol, "Fig. 1", a volatile oil, has been used since antiquity as a food flavoring agent, in the manufacture of fragrances, as a spice or condiment, in alternative or complementary medicine, as antibacterial-antifungal agent, as insecticide or insect repellant. In recent times, it is an ingredient in many brands of mouthwash and toothpastes. It also serves as a cough suppressant in cough syrups, incorporated in balms to relieve sores and pains, and as an additive in antiseptic liquids. [1]

"Silver Bird", "Fig. 2" a well known brand in Nigeria, has been for years used for both personal usage and large or small scale industrial production of some commodities. It is therefore imperative that some analysis (no matter how little) be done in order to ascertain the properties and consequently the economic values of the oil.

Previous research carried out, though not actually and specifically on Silver Bird, but on eucalyptol extracted from blue gum or *eucalyptus globules* (a specie of eucalyptus plant), in Algeria, in March 2012, reported that eucalyptol, after the physiochemical analysis, was found to be safe for use as a natural flavoring agent in food processing, as the results of the findings has proven to be close to the results obtained in other parts of the world. [1]

Silver Bird (eucalyptol), due to its increasing patronage by small and large scale industries, the research is therefore aimed at exploring its iodine and Saponification values, as the two are important parameters in quality assessment of an essential oil, hence a prerequisite to its industrial potentials. And it will also serve as a guide to local and international cosmetic and pharmaceutical products manufacturers and end users; for the price of a pharmaceutical, or any chemical, must have built into it not only the actual cost of production, but an element to pay for the years in which the product was being developed. [2]

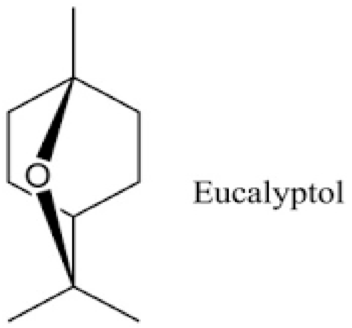

Figure 1: chemical structure of eucalyptol .

Figure 2: image of silver bird eucalyptol.

II. Experimental.

2.1 Sample Collection.

Sample was obtained from a local pharmaceutical chemist, eucalyptus oil (100%) of "Silver Bird" brand.

2.2 Iodine Value Determination.

The iodine value or "iodine adsorption value" or "iodine number" or "iodine index" in chemistry is the mass of iodine in grams that is consumed by 100 grams of a chemical substance. An iodine solution is yellow/brown in color and any chemical group in the substance that reacts with iodine will make the color disappear at a precise concentration. The amount of the substance thus required to keep the iodine solution yellow/brown is a measure of the amount of the iodine sensitive reactive groups.

2.2.1 Reagents:

- Deionised water
-Wij's solution

- 1,1,1-trichloroethane
- Iodine solution
- 10% Potassium iodide solution
- 0.1M Sodium thiosulphate solution

2.2.2 Procedure:

- Iodine solution: This was prepared by 13g of iodine crystals in a $1dm^3$ volumetric flask containing 25g of potassium iodide crystals, and was made up to the mark with deionised water.
- Standardizing iodine solution: 0.3g of primary standard arsenic (III) oxide (As_2O_3) was dried for an hour at room temperature and was dissolved in deionised water. Few grains of NaOH were added and 3.5g of sodium crystals were added, and was titrated against the iodine solution.

Equations of Reactions:

$$2OH^- + H_2O + As_2O_3 \longleftrightarrow 2H_2AsO_3$$
$$H_2AsO_3^- + H^+ \longleftrightarrow H_3AsO_3$$
$$H^+ + HCO_3^- \longleftrightarrow H_2O + CO_2$$
$$I_2 + H_3AsO_3 + H_2O \longleftrightarrow 2I^- + HAsO_4^{2-} + 4H^+$$

- 10% Potassium iodide solution: This was prepared by dissolving 10g of potassium iodide crystals in warm water and made up to the mark in $100cm^3$ volumetric flask.
- 0.1M Sodium thiosulphate solution: This was prepared by dissolving 25g of sodium thiosulphate crystals in warm water and made up to the mark in $1dm^3$ volumetric flask.

- Standardizing Sodium thiosulphate: Sodium thiosulphate solution was run from the burette into the iodine solution until its original yellow/brown color was pale yellow. Then a few drops of starch solution was added, thereby producing a dark brown coloration. Further drop by drop of thiosulphate solution was continued until it turns into a pale purple color and then disappeared.

Equation of Reaction:

$I_2 + 2Na_2S_2O_3 \longleftrightarrow 2Na^+ + 2I^- + Na_2S_4O_6$

- Analysis: 2mg of the oil was mixed with 20cm^3 wij's solution and 10cm^3 1,1,1-trichloroethane, it was then left in the dark for 30 minutes. Next, 15cm^3 of 10% potassium iodide solution and 10cm^3 of deionised water was added. This was then titrated against 0.1M sodium thiosulphate (VI) solution. 1cm^3 of 0.1M sodium thiosulphate solution = 0.01269g of iodine. The difference between a control titration and the titration with the oil present multiplied by this factor, gives the mass of iodine absorbed by the oil.

2.3 Saponification Value Determination.

Saponification value or "Saponification number", also referred to as "sap" in short, represents the number of milligrams of potassium hydroxide or sodium hydroxide required to saponify 1g of fat under the conditions specified.

$$\text{Number of moles} = \frac{\text{Mass of Oil}}{\text{Relative Atomic Mass}}$$

2.3.1 Reagents:

- 0.5M alcoholic potassium hydroxide solution
- 0.5M hydrochloric acid
- Phenolphthalein indicator
- Distilled water

2.3.2 Procedure:

- 0.5M alcoholic potassium hydroxide: This was prepared by dissolving 30g of potassium hydroxide in 20cm^3 of distilled water and final volume was made to 1 litre (1dm^3) using 95% ethanol. The resulting solution was allowed to stand for 24 hours, before decantation and filtration.
- 0-5M hydrochloric acid: This was prepared by measuring 21.25cm^3 of the concentrated HCl acid, and was gently poured into 50cm^3 of distilled water, and was later transferred into 500cm^3 volumetric flask and made up to the mark with distilled water.
- Phenolphthalein indicator: This was prepared by dissolving 0.1g of phenolphthalein powder in 60cm^3 of ethanol, and made up to the mark in a 100cm^3 volumetric flask.
- Analysis: 50mg of the oil sample was weighed into a conical flask, 250cm^3 of approximately 0.5M alcoholic KOH solution was added. The reflux condenser was attached and the flask was immersed in boiling water for one hour. 2 to 3 drops of phenolphthalein indicator was added after refluxing, and was titrated carefully with 0.5M hydrochloric acid.

III. Results and Discussion

3.1 Iodine Value.

3.1.1 Blank Titration.

Table 1: Blank Titration for Iodine Value.

	1st Titration (cm^3)	2nd Titration (cm^3)	3rd Titration (cm^3)
Final Volume	46.2	48.2	47.3
Initial Volume	03.3	05.5	04.5
Titre	42.9	42.7	42.8

Average Volume: Taking 1st, 2nd and 3rd titre values, we have: $\frac{42.9 + 42.7 + 42.8}{3} = 42.8\text{cm}^3$

3.1.2 Sample Titration.

Table 2: Sample Titration for Iodine Value.

	1st Titration (cm^3)	2nd Titration (cm^3)	3rd Titration (cm^3)
Final Volume	18.0	22.7	22.5
Initial Volume	03.5	08.4	08.0
Titre	14.5	14.3	14.5

Average Volume: Taking 1st and 3rd titre values, we have: $\frac{14.5 + 14.5}{2} = 14.5\text{cm}^3$

3.1.3 The Iodine Number.

To find the iodine number, the relation below is used:

$\frac{1.269\,(a - b)}{W}$, Where a = Blank Titre value = 42.8cm^3

b = Sample Titre value = 14.5cm^3 and W = weight of the sample = 2.0mg

Therefore, iodine number = $\frac{1.269\ (42.8 - 14.5)\ cm^3}{2}$

= $\frac{1.269\ (28.3)}{2}$ = $\frac{35.9127}{2}$ = 17.956

3.2 Saponification Value.

3.2.1 Blank Titration.

Table 3: Blank Titration for Saponification Value.

	1st Titration (cm^3)	2nd Titration (cm^3)	3rd Titration (cm^3)
Final Volume	35.9	35.7	40.4
Initial Volume	05.0	04.9	09.5
Titre	30.9	30.8	30.9

Average Volume: Taking 1st and 3rd titre values, we have: $\frac{30.9 + 30.9}{2}$ = $30.9cm^3$

3.2.2 Sample Titration.

Table 4: Sample Titration for Saponification Value.

	1st Titration (cm^3)	2nd Titration (cm^3)	3rd Titration (cm^3)
Final Volume	24.5	26.3	25.5
Initial Volume	03.0	05.0	04.0
Titre	21.5	21.3	21.5

Average Volume: Taking 1st and 3rd titre values, we have: $\frac{21.5 + 21.5}{2}$ = $21.5cm^3$

3.2.3 The Sap Number.

To find the sap number, the relation below is used:

$\frac{56\ (a - b)}{2W}$, Where a = Blank Titre value = $30.9cm^3$

b = Sample Titre value = $21.5cm^3$ and W = weight of the sample = 50mg

Therefore, sap number = $\frac{56\ (42.8 - 14.5)\ cm^3}{100}$

= $\frac{56\ (28.3)}{100}$ = $\frac{1584.8}{100}$ = 15.848mg KOH/g

3.3 Discussion.

At the end of the research, it was noted that both the results obtained from the iodine and saponification values, as recorded from Tables 2 and 4, were close to the ones obtained from previous research, hence proving the 100% purity of the product (Silver Bird).[1] It is also implying that Silver Bird eucalyptol has a very low potential in both large scale and local manufacturing, especially in soap production. It is therefore only useful as an important ingredient because of its medicinal, cleansing, and flavoring potentials.

IV. Conclusion

It was concluded that Silver Bird branded eucalyptol should be used only as an additive in cosmetic, pharmaceutical and foods industries, because it has proven to be 100% pure product based on the iodine and saponification values obtained. And that this research conducted is subject to minor errors due to differences in weather, handling of reagents and sample. It will be better if further research is conducted in order to confirm and explore the hidden potentials of the oil.

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