International Baccalaureate
Extended Essay in Chemistry

The Effect of Iodine Value on Soap Bar

How does the production of soap bars from 5g of oil types (rose/ avacado/ olive/coconut/ corn oil) with different iodine values affect the mass and INS value of soap?

Word Count: 4000

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Introduction

Ancient civilizations including the Babylonians, Egyptians, and Sumerians discovered the cleansing properties of natural substances and lead to the formation of soap. Early forms of soap were made by mixing animal fats or plant oils with alkaline substances such as ash or lye, resulting in a cleansing agent. Ancient Egyptians mixed animal and plant oils with alkaline salts to form similar substance to soap for bathing and washing. In ancient Rome, soap became an daily hygiene material for bathing and cleansing. During the Middle Ages, soap-making process evolved in Europe by the discovery of the chemical properties of fats and alkalis. The Industrial Revolution of the 18th and 19th centuries lead to mass production of soap. The invention of synthetic detergents in the 20th century offered new formulations and applications for cleaning and hygiene. Currently, soap-making has become a highly specialized industry, with a wide range of products such as traditional soap bars to liquid soaps, shower gels, and specialty formulations contributed to different preferences. From its humble origins in ancient civilizations to its modern-day incarnations, soap has an essential place in our daily routine, protecting us from bacterias. Until the emergence of Covid in 2019, critics highlighted the importance of personal hygiene. Constantly washing hands with soaps, avoiding touching public spaces, using a mask and cologne recommended for keeping hands hygienic. I tried several soaps with various aromas, quality, foaming and moisturuzing properties during pandemic.

Soup is composed of sodium salt of fats or fatty acids. It is formed by the action of alkali on these acids. Saponification is the soap making process which triglycerides are combined with a strong base (sodium hydroxide) to form fatty acid metal salts. The distribution of unsaturated and saturated fatty acid determines the hardness, aroma, cleansing, lather, and moisturizing abilities of soaps. Oils rich in saturated fats (like coconut oil) tend to produce a heavier bar, while those rich in unsaturated fats (like olive oil) yield a softer soap.

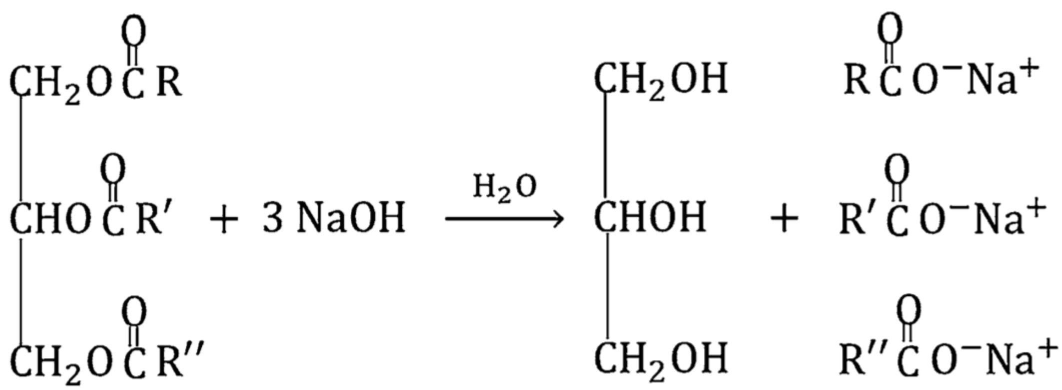
In the Journal of Surfactants and Detergents, “The Influence of Different Oils on the Properties of Soap Bars” was being published in 2014. Research focused on the effect of different oils including olive oil, palm oil and animal fats to the soap’s hardness, solubility, lathering ability and cleaning powder. It concluded that animal fats formed harder soap bars whereas vegetable oils produced moisturizing soaps. It influenced me to work on producing soap from plant oils.

I decided on a natural ingredient tailored to my skin type, avoiding harsh chemicals. Rather than using the synthetic oils present in commercial soaps, I prefered to use a variety of plant oils including rose, avacado, coconut, olive and corn oil to fragrance the soap bars. After formation of soap bars from 5 different oil, I followed titration process to calculate the iodine values. I weighted each soap bar on a digital balance and measured the corresponding mass to observe the relationship between iodine value and mass.

Saponification Process

Saponification is an exothermic chemical reaction that takes place when fats or oils (fatty acids) interact with lye, a base. During this process, the triglyceride components of fats interact with sodium hydroxide or potassium hydroxide, resulting in the formation of soap and glycerol. Various soap ingredients exhibit distinct characteristics and influence saponification process. Depending on the oil used (rose, coconut, olive oil e.t.c), soaps exhibit variation in lather, bar consistency, and cleansing ability. Saponification reaction:

Figure 1: General saponification reaction



Triglyceride + lye (base) = glycerol + fatty acid salt

Saponification Value

Oils and fats each have a "saponification value," indicating the amount of sodium hydroxide needed to completely saponify a particular oil. Through saponification process, the acid and the base are neutralized. The proportion of acid to base required for the saponification process is determined by the saponification value of each oil. Each type of oil whether it is plant or animal oil has a distinct saponification value based on its molecular structure.

Table 1. Average Theoretical Saponification Value

Oil Type	Saponification Value
Rose Oil	240 mg NaOH/g
Avacado Oil	190 mg NaOH/g
Olive Oil	190 mg NaOH/g
Coconut Oil	260 mg NaOH/g
Corn Oil	195 mg NaOH/g

Iodine Value

The iodine content signifies the amount of iodine in grams that reacts with 100 grams of a compound. The quantity of iodine consumed is directly proportionate to the number of double bonds present in fats and oils. Fats and oils contain both saturated and unsaturated fatty acids. Halogens form compounds when added across to double bonds of unsaturated fatty acids. The higher the iodine value, the more unsaturated fatty acids are present in the oil or fat. Soaps made from oils with higher iodine values tend to be softer and might lose mass more quickly when used because they can dissolve or wear away faster. Soaps made from more saturated fats (with lower iodine values) are usually harder and can hold their mass for a longer period during use.

Ins Value

Since the early 1900s the INS (Iodine Number Saponification) value is being used in the soap-making industry. It stems from the need to assess the properties of produced soap bars based on the oils and fats used in the formulation. Iodine value measuring the degree of unsaturation in fats and oils lead the discovery of Ins value. The equation "Saponification Value minus Iodine Value" gives the INS value for each type of oil. It can be used as a quick check to identify blends that saponify easily and make a hard soap suitable for high volume processing and packaging.

Hypothesis

Oils with higher iodine values having greater degree of unsaturation might form lighter bars cause they might lose mass quickier when dissolved or weared away.Consistently, oils rich in saturated fats with lower iodine values may produce heavier soap bars as they might hold their mass for a longer period during use.

Variables

Table 2. Variables

Type of variable	Variable	Method of controlling/ measuring variable
Independent Variable	Type of oil being used (rose, avocado, olive, coconut, corn oil)	5 different oils having different iodine values is being used in saponification and titration process.
Dependent Variable	Mass of soap bar (g)	Mass of each soup bar obtained is calculated by digital balance ±0.01g.
	INS Value	Ins value of each type of oil is being calculated by using their saponification and iodine value in an equation.
Controlled Variable	Amount of oil (g) used in titration method.	Constant amount 0.3g of oil is taken for each oil type by weighting them on a digital balance for iodine measurement.
	Amount of oil (g) used in saponification process.	5.000 g of each oil type was being used in the formation of soap bars.
	Constant Time Interval for each soap bar made by distinct oil type.	The solution allowed to react for 20 minutes at 60 °C under continuous stirring using magnetic stirrer with heating plate. Formed soap bars left for 1 day at room temperature.
	Apparatus / Reagents	Same apparatus and reagents in constant volumes is being used.
	Temparature (°C)	Experiment is being done in lab environment at constant room temparature.

Apparatus

-500.000cm³ Iodine Flask
-500.000cm³ Round flat bottom flask
-100.000cm³ measuring cylinder ± 1.000cm³
-250.000cm³ beaker ± 5.000cm³
-Soap mold
-Stirring hot plate
-Magnetic Stirrers
-Glass rod
-Thermometer ± 1.000cm³
-Clamp and stand
-Dropper
-Digital balance ± 0.100g
-50.000cm³ burette ± 0.100 cm³
-Magnetic Stirrers
-Lab Coat
-Safety Goggles
-Fume Hood
-Gloves
-Filter Paper

Reagents

5.000 g ± 0.001 Sodium hydroxide
1.500 ± 0.001 mL bromine solution
3.000 ± 0.001 g of potassium iodide solution
Glacial Acetic acid
6.200g ± 0.001 Sodium Thiosulfate solution
1.00g± 0.01 of Starch
20.00± 0.01 mL of Chloroform

Table 3. Reagent preparation

Reagents	How to obtain solution?
Hanu's Iodine Solution	6.800g of iodine dissolved in 412.000ml glacial acetic acid. Well powdered iodine kept in a round bottom flask. 412.000ml of glacial acetic acid added in small quantities and the flask is heated on a boiling water bath. The clear solution is decanted regularly followed by further addition of acid until all the iodine dissolved. The solution should be prepared in amber colored bottle.
Bromine Solution	A 1.5% solution is prepared by dissolving 1.500 ml of bromine in 100.000 ml of glacial acetic acid and preserved in amber colored bottle.
Potassium Iodide Solution	20.00ml of 15% solution is prepared in water by dissolving 3.000g of potassium iodide in 20.000mL of H_2O.
Sodium Thiosulphate Solution	Approximately 1N solution of $Na_2S_2O_3$ is prepared by dissolving 6.200g of $Na_2S_2O_3$, 5 H_2O in 250.000 ml of water. The solution is standardized against standard $K_2Cr_2O_7$ solution.
Starch İndicator	1.00g starch is made into paste with 10ml of distilled water. The suspension is poured into 90ml of boiling water and boil for 1-2min and cooled.

Safety Precautions

-Wear chemical-resistant gloves to avoid contact with highly corrosive chemicals like Hanu's Iodine Solution, Potassium Iodide Solution and Bromine solution. They may cause severe irritation, burns,long-term skin damage or absorption. Inhalation of bromine vapors can cause respiratory irritation.

-Wear lab coats to safeguard your clothing and skin against splashes, spills, or any contact with chemicals throughout the titration process. Shake the flask by ensuring all the iodine is expelled from the chloroform layer. Avoid direct skin contact with toxic and volatile materials such as iodine and chloroform. While working with them, consider using a fume hood or wearing a respirator to prevent respiratory problems.

- NaOH splash cause permanent eye damage and blindness. Put on goggles to avoid direct exposure to the lye solution and splashes.

-Sodium Thiosulphate Solution often considered as non-toxic, but may cause mild irritation to eyes and skin. Wear gloves and safety goggles when handling. Wash hands thoroughly after use. Store in a cool, dry place away from incompatible substances, like acids.

Environmental Concerns

I decided to produce bar form of soap instead of liquid state due to its sustainability. It is energy efficient and environmentally friendly to produce as it requires fewer resources. It comes with minimal or easily decomposable packaging which may last less to decompose in nature. Secondly, I decided on plant oils for the scent of the soap bars instead of using synthetic alternatives. When produced sustainably, they have several advantages including renewable sourcing, lower carbon emissions, biodegradability, and reduced toxicity. Coconut oil, olive oil, and avocado oil are non-toxic to humans, animals, and ecosystems unlike synthetic oils.

Furthermore, I handled iodine waste and other chemical reagents remained from the titration method as hazardous waste and treated to neutralize them before disposal. To prevent soil and air pollution, chemicals are disposed of in sealed containers designed for hazardous waste.

Methodology

Soap Preparation

1. Initially, 5.000 g of each oil type was weighed and transferred into 250.000cm^3 beaker ± 5.000cm^3. Oils in solid state such as coconut oil, melted by heating.

2. Next, 33% lye solution was prepared by dissolving the required weigh NaOH in a required amount of water. The amount of water was calculated using the following equation:

Weigh of water (g)= Weight of Lye × (100-33)/33. A 33% lye solution was chosen because it was found to be the optimum concentration suitable for a hard bar of soap.

3. The amount of sodium hydroxide used was calculated using the theoretical saponification value of each oil according to the formula:
   Amount of lye (g) =Weight of oil (g) × Saponification Value
   The dissolution of NaOH in water is highly exothermic. Therefore, the 33% lye solution should be allowed to cool down below 40°C before adding it to the oil.

4. The 33% lye solution was transferred to the oil and allowed to react for 20 minutes at 60 °C under continuous stirring using magnetic stirrer with heating plate. After the saponification reaction was complete, the formed soap was transferred to petri dishes and left for 1 days at room temperature. Afterwards, the formed soap was weighted and cut into different pieces for further analysis.

Determination of Iodine Value

1. 0.300g of the oil was dissolved in 20ml of chloroform into the flask.
2. The Hanu's Iodine Solution was prepared in amber colored bottle as explained in the table above.
3. 25.00 ml of Hanu's iodine solution was pipetted out and mixed with 5ml of bromine solution into the flask.
4. The flask was closed with stopper and kept it in dark for 30 minutes.
5. A fresh potassium iodide solution was prepared by dissolving 15.00g of KI free from potassium iodate in 100.000ml of water.
6. 20.00 ml of 15% KI solution was added and mixed constantly with magnetic stirrer.
7. The mixture was titrated by standard sodium thiosulphate solution using starch as an indicator with vigorous shaking to extract the iodine from the chloroform layer.
8. The titration was continued until the disappearance of blue color which means the end point has reached.
9. Conduct a blank using 20.00ml chloroform instead of fat or oil.
10. The flask must be shaken thoroughly throughout the titration to ensure that all the iodine is expelled from the chloroform layer.

Since 1ml of 1.0N $Na_2S_2O_3$=12.69 of Iodine, the iodine value was calculated using the formula below.

$$\text{Iodine value} = \frac{(V_1 - V_2)\ cm^3 \times N \times 12.69}{W}$$

Where,

V1 = Volume, in cm^3, of $Na_2S_2O_3$ solution required for the blank
V2= volume, in cm^3, of $Na_2S_2O_3$ solution required for the sample
N = normality of $Na_2S_2O_3$ solution (1.0N)
W =weight, in g, of the oil

Results

Table 4. Volume of Oils Raw Data

Type of Oil	Volume 1 (cm^3) ±0.01					Volume 2 (cm^3) ±0.01				
	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Rose	20.00	20.00	20.00	20.00	20.00	16.31	16.67	16.43	16.19	16.19
Avakado	20.00	20.00	20.00	20.00	20.00	18.10	18.10	17.86	17.86	17.86
Olive	20.00	20.00	20.00	20.00	20.00	18.21	17.86	17.98	17.98	17.98
Coconut	20.00	20.00	20.00	20.00	20.00	19.76	19.75	19.75	19.76	19.76
Corn Oil	20.00	20.00	20.00	20.00	20.00	17.26	17.14	17.26	17.02	17.02

Sample 1. Iodine Value Calculation for Rose Oil (Trial 1)

$$\text{Iodine value} = \frac{(V_1 - V_2)\ ml \times N \times 12.69}{W}$$

$$\frac{(20.00 - 16.31)\ cm^3 \times 1.0 \times 12.69}{0.300} = 156$$

Percentage Uncertainty for Rose Oil Trial 1

Uncertainty for V_2-V_1

20.00±0.01 - 16.31±0.01 = 3.69 ± 0.02

$\frac{0.02}{3.69}\times 100 = 0.54\%$

Uncertainty for weight of oil (0.300±0.001)

$\frac{0.001}{0.300}\times 100 = 0.33\%$

0.54% + 0.33%=0.87%

Table 5. Iodine Value Processed Data

Type of Oil	Iodine Value				
	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Rose Oil	156±0.87%	141±0.93%	151±0.89%	161±0.85%	153±0.85
Avocado Oil	80.4±1.4%	80.4±1.4%	90.5±1.3%	90.5±1.3%	85.5±1.3%
Olive Oil	75.7±1.5%	90.5±1.3%	85.5±1.3%	85.5±1.3%	84.3±1.3%
Coconut Oil	10.2±9%	10.6±8%	10.6±8%	10.2±9%	10.4±9%
Corn Oil	116±1.1%	121±1.0%	116±1.1%	126±1.0%	120±1.0%

Standard Deviation and Average Iodine Value Calculation for Rose Oil

The mean= $\frac{156+141+151+161+153}{5} = 152$

Standard Deviation= $\sum\sqrt{\frac{(xi-x)^2}{N-1}}$

Where;
xi = each individual value in the sample
x = sample mean (average)
N = number of data points in the sample
∑= sum of all the terms

Standard Deviation for Rose Oil

$$\sqrt{\frac{(152-156)^2+(152-141)^2+(152-151)^2+(152-161)^2+(152-152)^2}{4}}$$

= 6.61

Percentage Uncertainty for Rose Oil Mean

$\frac{\text{min}-\text{max}}{2\sqrt{\text{N}}} = \frac{160-141}{2\sqrt{5}} = 4$

$\frac{4}{152} \times 100 = 3\%$ 152± 3%

Table 6. Average Iodine Value of Oils

Type of Oil	Mean	Standard deviation
Rose Oil	152 ± 6%	6.61
Avocado Oil	85.5± 3%	4.50
Olive Oil	84.3± 4%	5.37
Coconut Oil	10.4± 0.86%	0.20
Corn Oil	120± 1.9%	3.71

INS Value Calculation for Rose Oil (Trial 1)

Ins Value= Saponification Value - Iodine Number

240 -152= 88

Table 7. Mass of Soap Bar

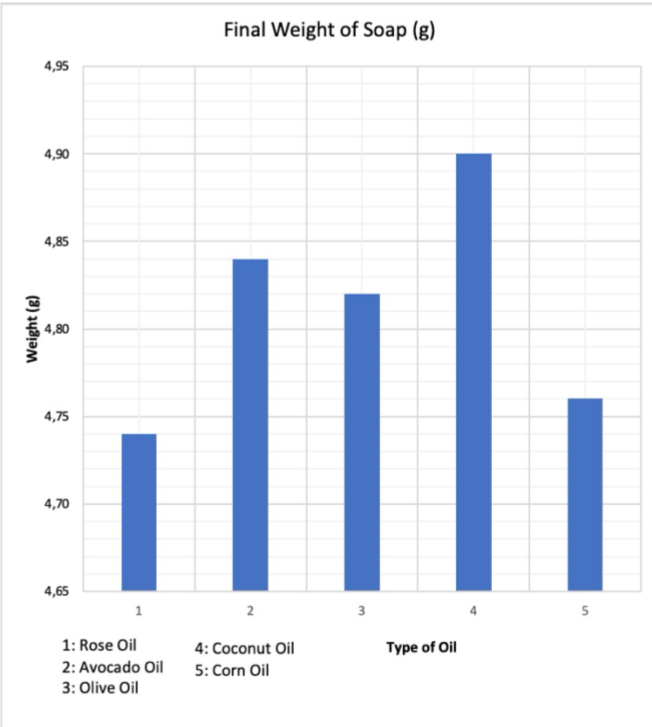
Type of Oil	Final Weight of Soap (g)	Saponification Value (NaOH/g)	Iodine Value	INS Value
Rose Oil	4.740g±0.001	240mg	152	88.0
Avocado Oil	4.840g±0.001	190mg	85.5	105
Olive Oil	4.820g±0.001	190mg	84.3	106
Coconut Oil	4.900g±0.001	260mg	10.4	250
Corn Oil	4.760g±0.001	195mg	120	75

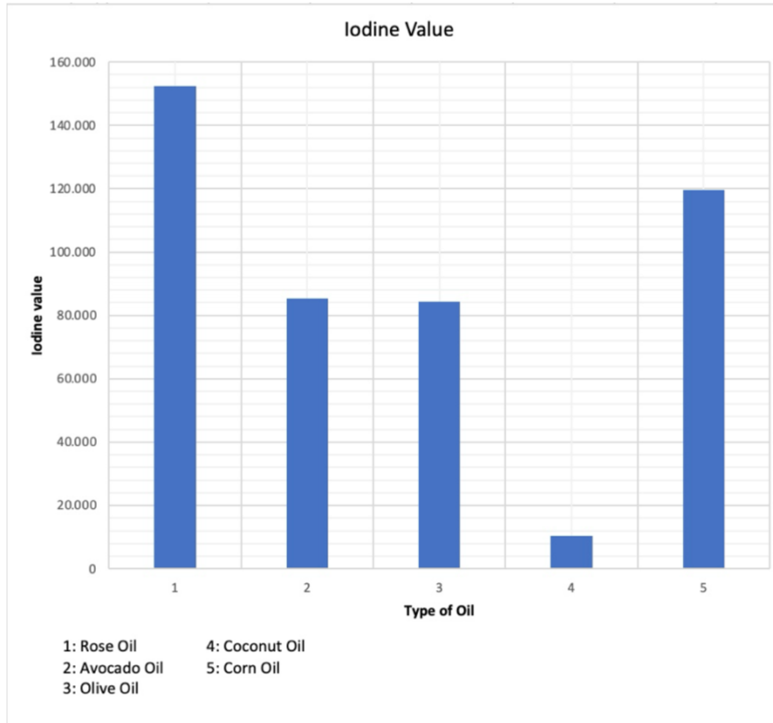
Conclusion

Throughout the experiment, the main concern is to compare the effect of oil type on the mass of the obtained soap bars and INS value. Mass of the final soap bar and its properties depends on the type of oil being used during saponification process. Each oil exhibit variation in lather, bar consistency, and cleansing ability when it interacts with a lye, base. I used a variety of plant oils including rose, avacado, coconut, olive and corn oil to fragrance the soap bars. Triglyceride components of oils interacted with sodium hydroxide, resulted in the formation of soap and glycerol after the saponification process completed. Formed soap bars transferred to petri dishes and left for a day at room temperature. Then, soap bars weighted on a digital balance and cut into pieces for further analysis. According to my calculations, final weight of obtained soap bars found to be 4.900g for coconut oil, 4.840g for avacado oil, 4.820g for olive oil, 4.740g for rose oil, 4.760g for corn oil, and 4.740g for rose oil. In background research, it is mentioned that "The higher the iodine value, the more unsaturated fatty acids are present in the oil or fat. Soaps made from oils with higher iodine values tend to be softer and might lose mass quickier while soaps made from more saturated fats with lower iodine values are usually harder and can hold their mass for a longer period during use." To determine iodine content, iodine mono-chloride is allowed to react with the oil in the dark. The excess of iodine released is titrated with standard sodium thiosulphate, using starch as an indicator. Iodine values found to be 152 for rose oil, 120 for corn oil, 85.5 for avacado oil, 84.3 for olive oil, and 10.4 for coconut oil on average. For iodine value calculation, 5 trials by using different volumes is done to minimize percentage error and their average is being calculated. Obtained iodine values and weighted mass of soap bars on digital balance confirmed that typically as iodine value increases, mass generally decreases. Highest iodine value with 152 and minimum weight with 4.740g belongs to rose oil while the lowest iodine value and maximum weight corresponds to coconut oil with 10.4 and 4.900g. Avacado and olive oil has moderate iodine values with 85.46 and 84.3. Their mass also measured to be close to each other with 4.840g and 4.820g. Lastly, corn oil has a relatively low iodine value and corresponding mass with 120 and 4.760g. The pattern of inversely proportional iodine value and mass among five different oils proved the accuracy of my hypothesis. It is also being verified that the distribution of unsaturated and saturated fatty acid influences mass. Oils rich in saturated fats like coconut oil tend to produce a heavier bar, while those rich in unsaturated fats like olive oil yield a softer soap as can be seen from table 7.

Figure 4. Macroscopic photos of rose, avacado, coconut , olive and corn oil-based soap bars

The reason of the approximate data between mass values for 5 different oil type does not source from corresponding iodine value or can not be seen as a personal error. There are several factors affecting the mass of a soap bar including additives, amount of lye used, water content, curing process and time. Therefore, the generalization between weight and iodine value is not based purely on oil type. A higher Iodine value doesn't absolutely mean the soap will be softer or have reduced weight; it's just a sign that it might.





Secondly, I investigated the relationship between the oil type used during the formation of soap bar and corresponding INS value. INS number refers to unit-less numerical value that explains the properties soap will have depending on the saponification and iodine value of the used oil. It illustrates the correlation between the oil type and hardness, conditioning, lather, iodine, cleansing properties of the desired soap bar. In this theory, a soap mixture with 160 INS will be a perfect bar of soap. The formula for calculating the INS value is (Saponification Value – Iodine Value). Saponification value is the amount of alkali (sodium hydroxide) needed to completely saponify a particular oil while iodine value measures the amount of iodine that bonds to the unsaturated bonds (double bonds) of the oils. The average of processed data is used for the INS value equation. Resulting INS values from the given saponification and iodine values are found to be 75.3 for corn oil, 85.8 for rose oil, 104.54 for avacado oil, 105.7 for olive oil, and 249.6 for coconut oil.

In the field of handmade soap, it is stated that soap formulas have an "ideal" INS value. It has been first popularized by Robert McDaniels in his book Essentially Soap published in 2000. A perfect soap formulation, according to him, should have an INS value of roughly 160. Based on this theory, with an INS of almost 260, coconut is at the top of the list.The following fats with INS values between 140 and 160 are tallow, cocoa butter, palm oil, and lard oil. Avocado oil, sweet almond, high-oleic sunflower, and olive oil are among the fats having an INS of 95 to 115. Rice oil, corn oil, pumpkin, regular sunflower, soybean, canola, hempseed, and flaxseed have INS from 45 to 85. According to my calculations, from highest to lowest, coconut, olive, avocado, rose and corn oils were located. Finding consistent values between the given ranges refers to the accuracy and scientific nature of my extended essay.

Discussion & Evaluation

Strengths

In saponification process, amount of sodium hydroxide and the extent of water in the reaction determines the formation of soap bar. A higher amount of NaOH will ensure complete conversion of the triglycerides into soap and glycerol whereas less lye usage may cause greasy or incomplete soap production. I used 33% lye solution because it was found to be the optimum concentration suitable for a hard bar soap. The amount of sodium hydroxide was calculated by multiplying the weight of oil by the theoretical saponification value of each oil. The water and lye contents of soap bars provided a desired structure which increase the yield and quality of the soap. They regained their form swiftly and were not sticky. There were not any loss of mass and no divisions when I cut them into pieces.

Accurate measurement for oil volumes is being done by filling graduated cylinder and repeating 5 times for each oil type. It is ensured that readings of the pipette or burette, as well as incorrect burette alignment during titration, can result in inconsistent findings.

Solutions are mixed constantly with magnetic stirrer to impede localized concentration differences and skewed results.

Weaknesses & Improvements

The endpoint in the titration is detected by the color change of the starch indicator. Endpoint is recognized by human eye which is a subjective approach as different individuals may interpret the endpoint differently. Although, I tried to avoid personal error in the determination of the disappearance of blue color, there may have been minor deviations. Instead pH meter could be used to identify the pH continuously as the titration proceeds. The pH curve shows a rapid change close to the endpoint, which enables to settle the exact point where the equivalence is reached.

Vigorus shaking during titration enhanced the potential of splashing or reagent loss, which end up in imprecise readings. When modest amounts of sodium thiosulphate solution as titrant introduced, excessive shaking caused the contents of the flask to overflow, lowering the amount of titrant accessible for the reaction. To reduce this risk, it is crucial to maintain a moderate and controlled level of agitation.

During saponification process, 5g of oil was being used in the formation of soap bar. Unlikely, iodine value is determined by dissolving 0.300g of each oil in 20ml of chloroform. Iodine value calculation could have done using 5g of oil in the titration method to avoid complexity in the thesis.

It is possible to calculate the saponification value of oils by titration method as well. Instead of following titration method, I used the theoretical saponification value of each oil type.

Further Investigation

To increase the scope of the experiment, different types of oils can be examined throughout saponification process.By altering the curing time and condition in various oil kinds, soap's durability and hardness can be investigated. Finally, to determine the best sort of oil for the soap-making process, quantitative tests on soap performance, such as foam stability, skin irritation, and cleaning ability can be performed.

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