"DETERMINATION OF ACTIVE DETERGENT CONTENT OF "Rin" POWDER" & "COMPARATIVE ANALYSIS OF THE CORRELATION OF IODINE VALUE FROM TWO DIFFERENT METHODS"

By

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DECLARATION

Work described in this thesis was carry out by my self the laboratory of Unilever Ceylon Ltd, under the supervision of H.I.S.C.Gunasekera (Laboratory manager) and Dr.Nirmali Wickramaratne (SUSL). A report on this has not been submitted to another University for another degree.

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Lovely dedicated to

Dear parents and sisters

ACKNOWLEDGEMENT

ABSTRACT

DETERMINATION OF ACTIVE DETERGENT CONTENT OF "Rin" POWDER

"Rin" is a popular detergent powder formulation in the Sri Lankan market. It has an Anionic Detergent (AD) as the active ingredient.

It is vital to maintain the level of the active ingredient at the optimum. This is an essential factor for the performance in laundering.

Therefore % AD contents of 30 Rin samples were determined according to a method described in the Unilever Methods of Analysis over a period of two months. The average % AD content was **15.1±0.63**. The Unilever specification of % AD in Rin is **14.0±1**.

During the period of two month the % AD level is higher.

COMPARATIVE ANALYSIS OF THE CORRELATION OF IODINE VALUE FROM TWO DIFFERENT METHODS

The iodine value (IV) is a measure of the unsaturation of fats and oils and is expressed in terms of the number of centigrams of iodine absorbed per gram of sample.

The IVs for Toilet Blend oil samples were calculated using two methods.

        1.Wijs method

        2.GLC method

Wijs method is based on the iodometric determination of the iodine absorbed to the (C=C) double bonds.

Iodine Value from the GLC method was obtained by a theoretical calculation using fatty acid profile.

The IVs of 30 Toilet oil blend samples were determined over a period of two months. The average % IV content from GLC Method was **34.88±2.466**, while the average IV from Wijs Method was **36.49±2.198**.

- According to the IVs from both methods

The Wijs method IV > GLC method IV.

- $y = 0.0959x + 33.392$

There is a positive correlation between two IVs.

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DETERMINATION OF ACTIVE DETERGENT CONTENT OF "Rin" POWDER

CHAPTER-01

1.0 Introduction

Use of powdered detergent in both hand washing and machine washing process is mainly popular among the consumers. Rin is a powdered detergent that has reached the consumer effective. Even though there are other competitive products in the market.

The Rin powder is being used as a good cleaning agent in Sri Lanka, it is a good example for NSD (Non-Soapy-Detergent) powder. As well as we can use the name surface-active agent.

Water has a high surface tension. Therefore it is so difficult for water to wet the fabric. These problems are overcome by lowering the surface tension of water considerably by the use of suitable wetting agent in appropriate concentration. Rin is a good wetting agent and ideally suitable for this purpose[1].

1.1 Objectives

Process control of anionic detergent level in Rin powder.

It is important to control the level of anionic detergent in Rin powder in order to keep up the quality of the product.

2.0 Literature Review

Among all surfactants, anionics are produced with the most important volumes; they are generally more cost-effective than the others and are found in almost all kind of cleaners.

By definition, the hydrophilic part of the molecule carries a negative charge; the ionised moiety can be a carboxylate, sulfate, sulfonate or phosphate.

Dealing with common anionic detergents, we can place these detergents into the following main groupings[3].

Alkyl Aryl Sulphonates: Linear alkyl benzene sulphonate would be the highest quantity used of any detergent in the world, and the alkyl aryl Sulphonates as a group would represent more than 40% of all detergent used.

They are cheap to manufacture, very efficient, and the petroleum industry is a starting point for the base raw material. The most important alkyl aryl condensate is DDB (dodecyl benzene). DDB is sulphonated to DDBSA (dodecyl benzene sulphonic acid), and this in turn is used as a detergent base, where it is neutralised with a base, such as sodium hydroxide, monoethanolamine, triethanolamine, potassium hydroxide, etc.

Chemical structures

$CH_3 - (CH_2)_x - CH(C_6H_5) - (CH_2)_y - CH_3$;

Linear alkylbenzene (LAB)

$CH_3 -(CH_2)_x - CH(C_6H_4SO_3Na) - (CH_2)_y - CH_3$

Sodium linear alkylbenzene sulfonate (LAS)

$R1 - C_6H_4 - SO_2 - C_6H_4 - R_2$ $C_6H_4 - C_3H_4R_1(R_2)$

Sulfone   Indanes

$C_6H_4-CHR_1-C_2H_4-CHR_2$

Tetralines

Long Chain (Fatty) Alcohol Sulphates

Made from fatty alcohol, and sulphated, these are used extensively in laundry detergents. They can be produced with varying carbon chain lengths, but a C_{12} - C_{18} alcohol sulphate is a good choice[3].

Other groups[2]: Are the olefin sulphates and sulphonates, alpha olefin sulphates and sulphonates, sulphated monoglycerides, sulphated ethers, sulphosuccinates, alkane sulphonates, phosphate esters, alkyl isethionates, sucrose esters.

(i). Alkyl Sulfonates

Chemical structures

CH_3- $(CH_2)_M$- $CH_2(SO_3Na)$

Primary sodium Alkyl Sulfonates

CH_3- $(CH_2)_M$- $CHCH_3(CH_2)_N(SO_3Na)$

Secondary sodium Alkyl Sulfonates

R-$(CH_2)_3$-CH=CH_2 + SO_3^- → R-$(CH_2)_3$- CH^+ - CH_2SO_3
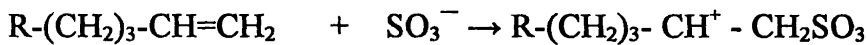
Alpha olefin sulfonation yields an intermediate zwitterion.

R-CH_2- R-CH_2-CH_2CH_2- ^+CH-CH_2-CH_2-$^+SO_2O^-$

⇓

R-CH_2-CH_2-CH-CH_2-CH_2

$$O \text{ — } SO_2$$
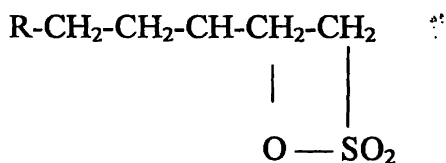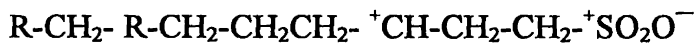
1,3 sultone formation from a zwitterion

$\pm 35\%$ $\begin{cases} R\text{-}CH_2\text{-}CH_2\text{-}CH\text{=}CH\text{-}CH_2\text{-}SO_3Na \\ R\text{-}CH_2\text{-}CH\text{=}CH\text{-}CH_2\text{-}CH_2\text{-}SO_3Na \end{cases}$

$\pm 65\%$ R-CH_2-CH_2-CH-CH_2-CH_2-SO_3Na

OH

4
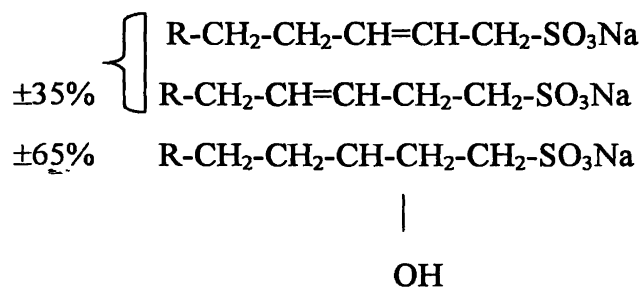
Products of alkaline hydrolysis of 1,3 sultone: Sodium alkenesulfonates and sodium hydroxy alkane sulfonate

(ii). Sulfo Fatty Acid Easters

Chemical structures

R—CH—(C=O)—O—CH₃   R—CH—(C=O)—O—R
    | |

SO₃Na   SO₃Na
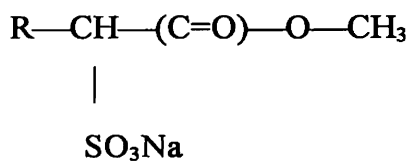
Methyl ester of α-Sulfo fatty acid,   Alkyl ester of α-Sulfo fatty acid,

Sodium salt   sodium salt

(iii). Fatty Acid Isothionates and Taurides

Chemical structures

R—CO—Cl   +

HOCH₂—CH₂—SO₃Na   →   R—CO—OCH₂—CH₂—SO₃Na + HCl

Fatty acid isothionate

R—CO—Cl   +   NaOH

NHCH₂—CH₂—SO₃Na   → R—CO—NCH₂—CH₂—SO₃Na +NaCl

| -H₂O |

CH₃   CH₃

Tauride
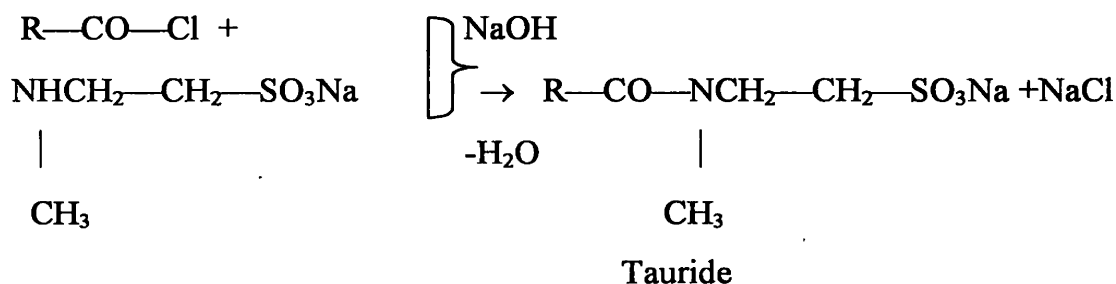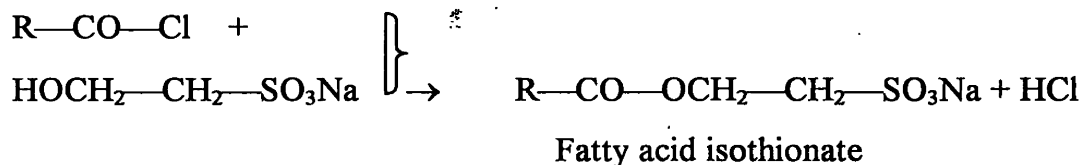
(iv). Phosphoric Acid Easters and Salts

Chemical structures

3ROH + P₂O₅ → RO—P=O(OH)₂   +   RO—P=O(OH)

|

OR

Alkyl Phosphoric Easter   Dialkyl Phosphoric Easter

R—(OCH$_2$CH$_2$)$_n$—O—P=O(ONa)$_2$

$$R—(OCH_2CH_2)_n—O \atop R—(OCH_2CH_2)_n—O} P=O(ONa)$$

Sodium ethoxylated alkyl phosphate

Sodium di (ethoxylated) alkyl phosphate
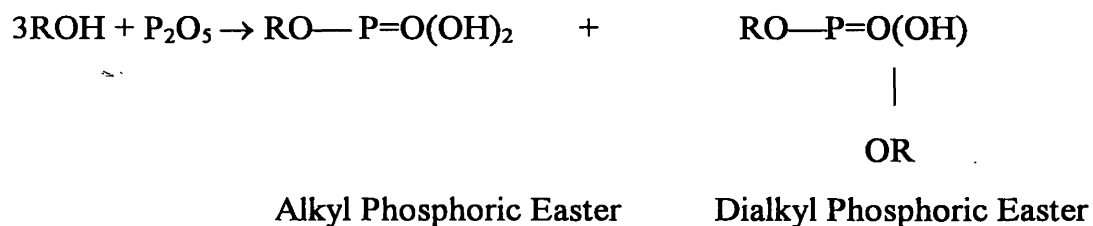
3.0 Materials and Methodology[4]

3.1 Samples collecting

Rin samples for the studies were collected from Quality Assurance Department.

3.2 Apparatus and Reagents

- Standard laboratory equipment, including. -Titration vessel.- 100 ml Tall, ground-glass stoppered cylinders, diameter 2.5 cm, height 25 cm, were used.

- Hyamine 1622, Mixed indicator. - Stock solution, Acid indicator solution, Chloroform Phenolphthalein.

3.3 Procedure

Standardisation of 0.004M Hyamine 1622

An aqueous solution of Sodium Lauryl Sulphate (SLS) of exactly known concentration was titrated with the Hyamine 1622 solution in a two phase water-chloroform system. A mixture of cationic dye (dimidium bromide) and an anionic dye (disulphine blue V) as an indicator. The SLS forms a salts with the cationic dye which dissolves in chloroform layer to give this layer a red-pink colour. The end point is reaches when the Hyamine cation displaces the dimidium cation from the chloroform soluble salt and the pink colour leaves the chloroform layer and the dye passing to the aqueous phase.

Method

An accurate weight (3.5- 5g) of Rin powder was taken into the beaker and dissolved in distilled water by heating using a hot plate (The Rin powder sample should be contained about 0.24 milliequivalents of Anionic active matter). The dissolved sample was transferred to a 500 ml graduated flask and diluted to a volume 500 ml with water, and mixed well.

10 ml sample from the prepared solution was pipetted into the titration vessel, a few drops of phenolphthalein was added and if required, it need to be neutralised with 0.1N NaOH solution to a faint pink colour. These following solutions were added sequentially 10 ml of distilled water and 10 ml of Acid Indicator, 15 ml of chloroform. The stopperd vessel was thoroughly shaken and titrated with standardised 0.004M 1622 Hyamine solution. In the titrant, the titration was run 0.5-1.0 ml at a time to within about 1ml of the end point and one-drop was finally added at a time. The titration vessel was thoroughly shaken after each addition of the titrant. The volume of the titrant used for the titration was recorded at the end point, this was the first point at which the pink colour was completely discharged from the chloroform layer, which was then a faint greyish blue. A blue-grey or blue colour indicates that the end point has been passed. The above procedure was repeated for 30 days process controlling of Rin powder samples.

CHAPTER-04

4.0 Results

Calculations. -

Weight (g) of the sample =W

Molarity of Hyamine 1622 solution =M

Mean molecular weight of anionic active matter =Q

Volume (ml) of aliquot $=V_1$

Volume (ml) of Hyamine 1622 used for titration$=V_2$

% Total anionic active matter $=(V_2 * M * Q * 100)/(W * V_1)$[4]

E.g. W=(5g * 10ml)/500ml =0.01g, M=0.004, Q=342g,V_1=10ml,V_2=12.1ml

% Total anionic active matter = (12.1 * 0.004 * 342)/(0.01* 10)

% Total anionic active matter =16.60.

(i). % AD level in Rin.

Table (i)- Used for % AD level during the period of two month.

Production Date	% Anionic active matter (AD±1)
14/09/2002	16.6
16/09/2002	14.8
17/09/2002	14.8
18/09/2002	16.9
26/09/2002	14.7
28/09/2002	14.9
30/09/2002	14.8
02/10/2002	14.8
03/10/2002	15.8
05/10/2002	14.1
14/10/2002	15.0
15/10/2002	15.9
16/10/2002	15.6
17/10/2002	15.2
23/10/2002	15.4
26/10/2002	14.8
28/10/2002	15.0
29/10/2002	15.1
30/10/2002	14.8
31/10/2002	15.4
01/11/2002	15.2
04/11/2002	15.4

05/11/2002	14.0
06/11/2002	14.2
08/11/2002	14.9
12/11/2002	15.1
13/11/2002	15.2
15/11/2002	14.8
16/11/2002	14.4
20/11/2002	14.8

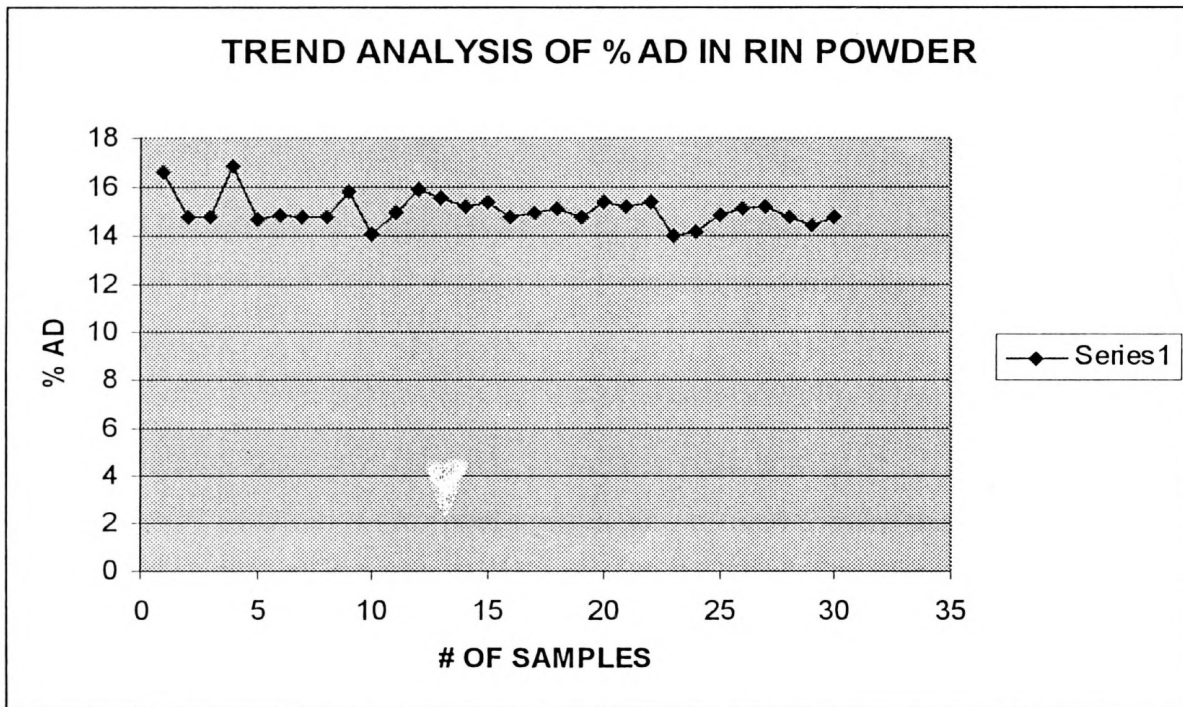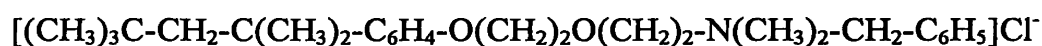**Average % AD =15.1 ±0.63**

(ii). Trend Analysis of % AD.

Fig (a)-Used for Trend Analysis of % AD in Rin powder.

5.0 Discussion[4]

- Hyamine 1622 is the commercial name for the benzethonium chloride, it has positive charges that's by used for the titration's to find AD.

    Chemical Structure of the Hyamine 1622

    $[(CH_3)_3C-CH_2-C(CH_3)_2-C_6H_4-O(CH_2)_2O(CH_2)_2-N(CH_3)_2-CH_2-C_6H_5]Cl^-$

- The sample to be taken may be determined by using the following formula:-

    Sample size (g)= 0.24 Q/Al.

Where Q is the mean molecular weight of the anionic active matter and Al is the expected total anionic active matter (%) of the sample.

Sample size will determine the volume of the titrant used, so we can reduce the chemical cost.

- When the Rin powder was dissolved in water, it was not totally dissolved in the water, but our expected % AD was got, So we can conclude that, all the ions are in liquid phase.

- An aqueous solution of the Rin powder sample is treated with cationic active solution (Hyamine 1622) in a two-phase water-chloroform system using a mixture of cationic dye (dimidium bromide) and an anionic dye (disulphine blue V) as indicator. The anionic surfactant forms a salt with the cationic dye, which dissolve in the chloroform layer and gives this layer a red-pink colour. The end point is reached when the Hyamine 1622 cation displaces the dimidium cation from the chloroform soluble salt and the pink colour leaves the chloroform layer as the dye passes to the aqueous phase. Hyamine added in excess forms a salt with the anionic dye- disulphine blues V- that dissolves in the chloroform layer and colours it blue.

CHAPTER-06

6.0 Conclusion

During the period of two month the % AD level is higher.

REFERENCES

1. Handbook for soap Detergent and Glycerine, pages 117-118. (By K.C.Dhingra)

2. Handbook of detergents (surfactant science series), part A, Volume 82, pages 8-21. (Edited by Guy Broze)

3. http://www.chemistry.co.nz/detergent_class.html.

4. Unilever Methods of Analysis (UMA).

COMPARATIVE ANALYSIS OF THE CORRELATION OF IODINE VALUE FROM TWO DIFFERENT METHODS

CHAPTER-01

1.0 Introduction

Unilever Ceylon LTD is one of the leading soap manufacturing company in Sri Lanka. There is a range of different soap brands and Toilet brand is one of most popular brand in Sri Lanka. Toilet blend is used to produce the Toilet soaps. There are lot of important parameters which are measure the quality of Toilet blend. The iodine value is one of the important parameter.

The iodine value (IV) is a measure of the degree of unsaturation of fats and oils and is expressed in terms of the number of centigrams of iodine absorbed per gram of sample. The IV serves as an indicator of relative hardness or softness of fats & resulting fatty acids & soap products. In the most general sense, higher levels of unsaturation, reflected by higher iodine values, indicate a tendency toward softer material & subsequently, softer soap products. Lower IV indicating less unsaturation, will exhibit a higher melting point and will result in firmer soap than will lard[2].

Toilet Soaps Oil blend is being mainly used to prepare Toilet Soaps such as Lux, Rexona, Pears Baby, Lifebuoy Gold. In the preparation of Toilet Soaps the Iodine Value is an important parameter in the determining the softness associated with Toilet blend. The IV is going to be determined using two methods, the GC and Wijs method. The IV is depend on the number of double bonds.

Since there are two accepted methods. It is important to find at the agreement of results obtained by both methods. Therefore my study involve in comparison of results obtained for the iodine value from GLC and Wijs method.

1.1 Objectives

Minimise the cost of analysis & improve accuracy of IV determination.

This study also focus on minimising the cost and improvement of the accuracy of these methods.

CHAPTER-02

2.0 Materials and Methodology

2.1 Samples Collecting

Toilet blend samples were collected from the Bulk Material Department. (30 samples)

2.2 Iodine value (IV) Analysis

Iodine values were analysed using two methods.

          1.Wijs method

          2.GLC method

2.2.1 Wijs method[1]
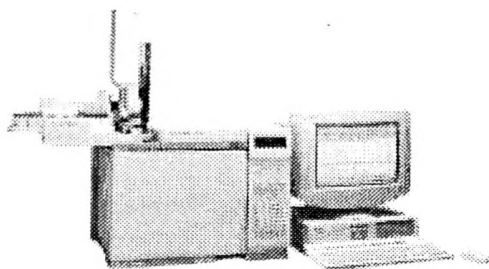
Apparatus and Reagents

- Glass stoppered iodine flask 500 ml, Pipette 20 ml, Burette, Filter paper.

- 0.2N ICl, CCl_4/$CHCl_3$ (Carbon tetrachloride (or) chloroform), 0.2N $Na_2S_2O_3$, 10% Potassium iodide (KI) solution, Starch solution.

2.2.2 GLC method[3]

Apparatus and Reagents

- Gas chromatograph, Recorder, Integrator (or) Calculator, Syringe Chromatographic column.

Fig(b)-Used for Example to Gas chromatograph.

- 125-ml flat bottom boiling flasks, Water-cooled condensers.

- Gases (He, H2, Dry Air), Reference standards.
- BF$_3$-Methanol, 0.5N methanolic sodium hydroxide, Sodium chlorite (NaCl) saturated solution in water, Isooctane.

2.3 Procedure

Standardisation of sodium thiosulphate solution[5]

The weight of 0.20 - 0.25g (W g) of well dried $K_2Cr_2O_7$ was dissolved in distilled water. 5ml of conc. HCl & 25ml of 10%KI were added and Kept in a dark place for half-hour. Using starch as indicator, the sample was titrated with prepared sodium thiosulphate solution(Vml).Carryout three determination to get an average figure.

1.Wijs method[2]

The prescribed amount of sample was accurately weighed into a iodine flask. Weight of sample can be determined as follows.

Weight = 20/Expected IV

The weighed sample was dissolved in an appropriate amount of solvent $CCl_4/CHCl_3$ (10 ml). Twenty ml of Wijs solution and 10ml of 10% KI were accurately added and mixed well, and left in the dark room for about 30-60 minutes. Distilled water was finally added and titrated with standardised 0.2N $Na_2S_2O_3$ solution, with constant and vigorous shaking. The starch indicator was added towards the end of the titration i.e. when the solution was turned to pale yellow (straw colour). Simultaneously a blank titration was prefered using the same quantity of reagents at the same time under same conditions. The above procedure was repeated for 30 different samples.

2. GLC method[3]

The oil (filtered oil) was introduced into flat bottom boiling flask (Accurate weighing is not required about 8 drops). The specified amount of (5-ml) 0.5N Methanolic sodium hydroxide was added and a boiling chip was added. A condenser was attached and the mixture was heated on a steam bath until the fat globules go into solution. This step should be taken 5-10 minutes. The specified amount (3-ml) of BF_3-Methanol was added through the condenser and boiled for two minutes. The 5-ml of isooctane was finally added through the condenser and boiled for one minute longer. The flask was removed from heat and condenser. The enough amount of saturated salt solution was added to float the isooctane solution of the methyl esters into the neck of the flask. About 1ml of the isooctane solution was transferred into a test tube. The isooctane solution was then injected directly in a gas chromatograph. The above procedure was repeated for 30 different samples.

CHAPTER-03

3.0 Results

1.Wijs method[2]

Calculations. -

The factor was used N=0.1916 from 11/10/2002 to 28/11/2002

N=0.1939 from 5/12/2002 to 31/12/2002

Weight of sample in grams = W,

Volume of thiosulphate used for sample = V_S,

Volume of thiosulphate used for blank = V_B,

Normality of thiosulphate = N,

$$IV = [(V_B-V_S)*N*126.9*100]/[W*1000]$$

$$IV = [12.69*N*(V_B-V_S)]/W$$

E.g.

V_S=18.9 ml, V_B=24.5 ml, W=0.3452g, N=0.1939

IV = [12.69*0.1939*(24.5 -18.9)]/ 0.3452

IV=37.6

2. GLC method

Calculation. -

(i). Identification of peaks[4]

The reference standard mixture of known composition was analysed under the same operating conditions as those employed for the sample and measure the retention distances (or retention time) was measured. A graph was Constructed output showing the

logarithm of the retention time as a function of the number of carbon atoms of the acids. Under isothermal conditions, the graphs for straight chain esters of the same degree of unsaturation should be straight lines. The peaks of the sample from chromatogram was identified by comparing the retention time of the standard mixture.
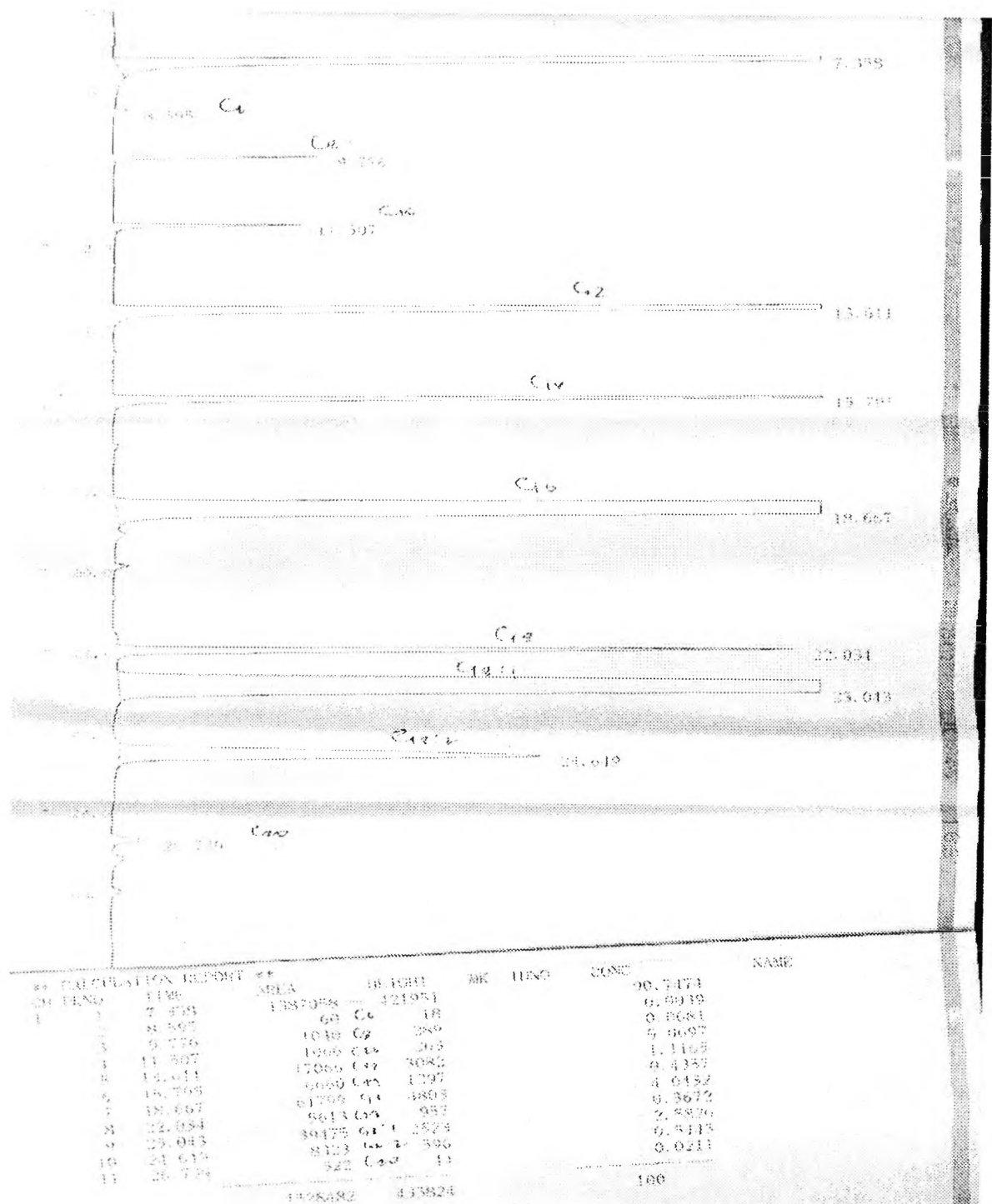
E.g.

Fig (c)-Used for Gas Chromatogram.

Table (ii)-Used for IV calculation from GLC method[5].

FATTY ACID COMPOSITION BY GLC				
	CALCULATED A.IIIb.11-13		AS PER UMA	
SAMPLE	Toilet Blend		9/12/2002	
SOURCE	BMD Tank 3			
CONDITIONS				
Initial Temperature:100 0C/ Rate: 09 0C per min/Split ratio: 1:100				
Final Temperature:200 0C/Attenuation: 1/Range :2/				
METHOD OF ESTERIFICATION :			BF$_3$ Method	
ANALYST	IG			
Component in Ref. Mixture	No of C atoms	AREA	% by mass	
Caproic acid	C$_6$	60	0.042	
Caprylic acid	C$_8$	1040	0.735	
Capric acid	C$_{10}$	1066	0.754	
Lauric acid	C$_{12}$	17066	12.067	
Miristic acid	C$_{14}$	6660	4.709	
Miristoleic acid	C$_{14:1}$	0	0.000	
Palmitic acid	C$_{16}$	61799	43.698	
Palmitoleic acid	C$_{16:1}$	0	0.000	
Stearic acid	C$_{18}$	5613	3.969	
Oleic acid	C$_{18:1}$	39475	27.913	
Linoleic acid	C$_{18:2}$	8323	5.885	
Arachidic acid	C$_{20}$	322	0.228	

Linolenic acid	C_{18}	0	0.000	
.		141424	100.000	
		%		
	IV	35.79		

Table (iii)-Used for IV calculation from GLC[5].

Formula			Mol Wt.	IV	
C	H	O			
6	12	2	116	0.00	0.00
8	16	2	144	0.00	0.00
10	22	2	174	0.00	0.00
12	24	2	200	0.00	0.00
14	28	2	228	0.00	0.00
14	26	2	226	0.00	0.00
16	32	2	256	0.00	0.00
16	30	2	254	99.92	0.00
18	36	2	284	0.00	0.00
18	34	2	282	90.00	25.12
18	32	2	280	181.29	10.67
20	40	2	312	0.00	0.00
18	30	2	278	273.88	0.00
					35.79

(i). IVs from both method.

Table (iv)-Used for IVs from both method.

| Production Date | IV | |
	GLC Method	Wijs Method
11/10/2002	31.04	34.25
14/10/2002	31.60	33.63
14/10/2002	35.65	35.76
16/10/2002	34.85	34.70
25/10/2002	35.38	36.77
25/10/2002	36.09	38.48
28/10/2002	34.77	36.31
29/10/2002	35.77	37.07
30/10/2002	34.56	35.49
02/11/2002	36.61	37.07
04/11/2002	28.59	31.02
04/11/2002	27.81	32.03
06/11/2002	36.43	36.85
07/11/2002	36.22	36.86
14/11/2002	36.21	36.41
15/11/2002	36.41	37.99
21/11/2002	34.86	37.42
24/11/2002	34.66	35.53
28/11/2002	34.71	35.46
05/12/2002	37.13	37.02
06/12/2002	36.53	34.20
09/12/2002	35.79	39.92

10/12/2002	35.14	36.06
11/12/2002	35.25	35.96
12/12/2002	35.69	38.07
13/12/2002	32.66	37.25
13/12/2002	33.35	40.74
17/12/2002	35.39	37.04
23/12/2002	37.65	38.68
31 12/2002	39.57	40.76

Average IV from GLC method =34.88±2.465752

Average IV from Wijs method =36.49±2.197836
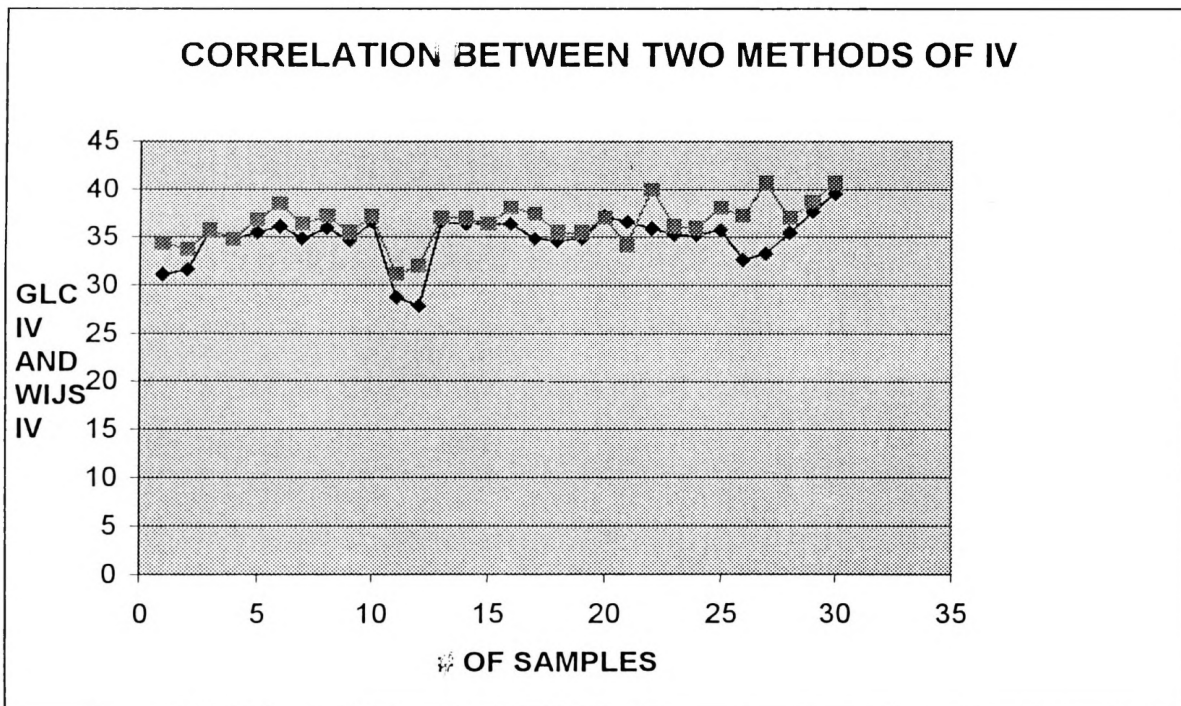
(ii). Trend Analysis of IVs.

Fig (d)-Used for Trend Analysis of IVs.

Correlation Co-efficient = 0.7070,

y = 0.0959x + 33.392

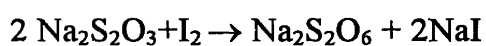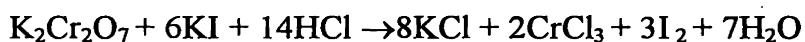# CHAPTER-04

4.0 Discussion

In Wijs method, the sample to be investigated is treated with an ICl solution (Wijs solution). The I_2 is liberated from excess ICl and KI and is titrated with standardised thiosulphate solution.[3]

$$ICl + I_2 \rightarrow 3ICl$$

$$ICl + KI \rightarrow I_2 + KCl$$

$$I_2 + 2Na_2S_2O_3 \rightarrow Na_2S_4O_6 + 2NaI$$
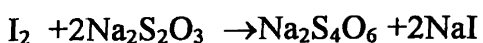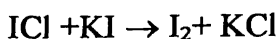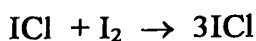
Standardisation of the sodium thiosulphate solution is very important in Wijs method. Here the strength is obtained to four decimals. If the strength is varies slightly, the iodine value also changed.

$$K_2Cr_2O_7 + 6KI + 14HCl \rightarrow 8KCl + 2CrCl_3 + 3I_2 + 7H_2O$$

$$2\,Na_2S_2O_3 + I_2 \rightarrow Na_2S_2O_6 + 2NaI$$

Normality = $(1000*W)/(49.03*V)$ [5]

- Amount of sample used is dependent on its iodine value and may be determined from the following table[5].

IV	Weight (g) of the sample
0-5	3.0
5-20	1.0
20-60	0.34
60-80	0.25
80-130	0.15
> 130	0.10

- Before weighing the sample it should be filtered through filter paper to remove any solid impurities and the last traces of moisture. The mechanical stirring was very satisfactory for agitating during the addition of thiosulphate. Then only we can get the sharp end point. And also we should keep the sample with wijs solution in dark room, because of wijs solution is sensitive to temperature, moisture, and light, for 1 hour at room temperature for complete absorption of iodine[4].

In GLC(calculated) method, Methyl esters of fatty acids are separated and determined quantitatively by GLC using a packed column. The method is applicable to methyl esters of fatty acids having 8 to 24 carbon atoms and to animal fats, vegetable oils, marine oils and fatty acids after their conversion of methyl esters. The method permits quantitative separation of mixtures containing saturated & unsaturated methyl esters. The conditions specified in this method are not suitable for determining epoxy (or) oxidised fatty acids (or) fatty acids that have been polymerised[3].

- So preparation of methyl esters (which due to their greater volatility) of long-chain fatty acids is very essential for further analysis by gas liquid chromatography method.

- In this method, if polyunsaturated components with more than three double bonds are present, they may decompose in a stainless-steel column.

- The required reaction time appears to increase as both the carbon number and degree of unsaturation increase. For longer chain and more unsaturated fatty acids, a general observation is that the reaction should be allowed to proceed just until there is no visual evidence of oil globules in the reaction mixture.

- The methyl esters should be analysed as soon as possible.

5.0 Conclusion

- According to the IVs from both methods

The Wijs method IV > GLC method IV.

- $y = 0.0959x + 33.392$

There is a positive correlation between two IVs.

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