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Statistical Analysis as a Tool in the Assessment of Detergents Produced from Seed Oils

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Abstract
Detergents are materials whose solutions aid in the removal of dirt or other foreign matter from contaminated surfaces. Until the 1940s, soap was the only important detergents. Today, soap is but one of a great many detergent products. The primary ingredient used in detergent manufacture is often called surface active agent or surfactant because it acts upon a surface. Detergents were produced from neat, benzene-modified and esterified seed oils; their properties were compared with those of OMO using statistical analysis (ANOVA). The analyses showed that: rubber seed oil is better than cherry seed oil in the production of detergent, esterification of the oil improved the detergent action and unmodified rubber and cherry seed oils produced soap and not detergent.
Keywords: Detergents, soap, statistical analysis, ANOVA, seed oil, and esterification

1. Introduction

Fats and oils resemble waxy esters but differ in that they are derivatives of glycerol, a tri-hydric alcohol. One of the areas where a mineral oil derivative, e.g. paraffin (both wax and oils) has been put to use is its modification for laundering application just as oils have been extensively exploited for use in soap manufacture [(1)]. During World War II the shortage of fats, from which soap was made, spurred the development of soapless or synthetic detergents, primarily in the United States. After the war the need for new types of detergents for automatic washing machines accelerated the trend [(2)].

Detergents are materials whose solutions aid in the removal of dirt or other foreign matter from contaminated surfaces. Until the 1940s, soap was the only important detergents. Today, soap is but one of a great many detergent products. The primary ingredient used in detergent manufacture is often called surface active agent or surfactant because it acts upon a surface. A common feature of detergent formulation is that the component surfactants are comparatively large molecules (molecular weight of over 200). One part of the molecule is soluble in organic material, and the other part is soluble in water [(2)].

Propylene tetramer (PT) benzene sulfonate held almost undisputed sway as the major ingredient used in washing operations till the early 1960s. Around this time it was noted, however, that sewage treatment problems were arising. The amount of foam on rivers was increasing and where water was being drawn from wells located close to household discharge points, the water tended to foam when coming out of the tap. This was attributed to the fact that propylene-based alkyl benzene sulfonates are not completely degraded by the bacteria naturally present in effluents, and was further narrowed down to the fact that it is the branched chain formation of the alkyl benzene which hinders the attack by the bacteria [(3)]. However, fatty acid sulfonates were found to degrade very easily. Since all naturally occurring fatty acids from which fatty alcohols are produced are of the straight-chain variety (as also are the Ziegler alcohols which started appearing in commercial quantities at about this time), it seemed possible that straight-chain alkyl benzene might be degradable [(3)]. Methods of test were developed and it was, in fact, proved that linear alkyl benzene (LAB) is biodegradable.

Germany introduced legislation prohibiting the discharge of non-biologically degradable material into sewer systems. In the USA detergent manufacturers agreed voluntarily to switch over from PT benzene to linear alkyl benzene by June 1965. In the United Kingdom a similar type of "gentleman's agreement" was entered into [(3)].

2.0 Partinent Issues in the Experimentation

The change to linear alkyl benzene gave some rather surprising results. It was found that the detergency in a

heavy-duty formulation using linear alkyl benzene sulfonate was approximately 10 percent better than when using PT benzene sulfonate [3]. Solutions of the neutralized sulfonic acid had a lower cloud point, and their pastes and slurries had a lower viscosity. The first two results were obviously advantageous and a lower viscosity in slurries had an advantage when the product was spray-dried to a powder, but when the linear alkyl benzene sulfonate (LABS) was sold as a liquid or paste detergent, this lower viscosity had to be overcome as sales appeal was lost. However, the manufacture of powders based on LABS posed some problems; powders became sticky and lost their free-flowing characteristics, whether made by spray-drying or one of the other methods.

2.1Target/Focus

The aim of this study is to use statistical methods in the analysis of the properties of fully soluble and biodegradable detergents from renewable and naturally available rubber seed oil in place of the linear alkyl compound that is obtained from petroleum sources which are finite natural resources. Screening of various seed oils for suitability as potential component in detergent formulation and production of detergents using neat, benzene-modified, and esterified rubber seed oils. Assessment of the properties such as pH, lathering ability and cleansing power of the produced detergent using statistical methods and comparison of the performance with those of linear alkyl benzene sulfonate and a commercially available detergent using Analysis of Variance.

3.0 Materials and Sources

Tables 1: Material Sources and Characteristics

Material	Sources	Characteristics
Rubber Seed	Locally sourced from Rubber Research Institute of Nigeria (RRIN), Iyanomo, Edo State	Base Material from which the required rubber seed oil was extracted
Rubber Seed Oil	Extracted from the seed at the Polymer Processing Workshop, Department of Polymer Tech, Auchi Polytechnic, Auchi	Contains Unsaturated Fatty Acid (Linoleic Acid). Modified to be used as surfactant $CH_3(CH_2)_4CH=CHCH_2CH=CH(CH_2)_7COOH$
Cherry Seed	Locally sourced from Uromi, Edo State	Base Material from which the required cherry seed oil was extracted
Cherry Seed Oil	Extracted from the seed at the Polymer Chemistry Laboratory, Department of Polymer Tech, Auchi Polytechnic, Auchi	Contains Unsaturated Fatty Acid (Ricinoleic Acid) 1-2-hydroxy-octadec-9-enoic a1-2-hydroxy-octadec-9-enoic acid. Acid. A rare source of an 18-carbon fatty acid with one double bond. Modified to be used as surfactant
Benzene	Juliros Nigeria Enterprises, 21 B Evbiemwen Street, off Wire Road, Benin City.	A colourless liquid used to modify rubber seed oil. Melting point (5.5^{o}C), b. point (80.1^{o}C), density (0.8765g/cm^3). Modifies oils.
Sulphonic Acid	Juliros Nigeria Enterprises, 21 B Evbiemwen Street, off Wire Road, Benin City.	A clear, colourless, odourless, liquid. Melting point (10^{o}C), boiling point (337^{o}C), density (91.84g/cm^3) Used for the sulphation of the surfactant.
Sodium hydroxide (caustic soda)	Juliros Nigeria Enterprises, 21 B Evbiemwen Street, off Wire Road, Benin City.	White solid, hygroscopic. Melting point (318^{o}C), boiling point (1388^{o}C), density (2.13g/cm^3) Used for neutralization.
Sodium Carbonate (soda Ash)	Juliros Nigeria Enterprises, 21 B, Evbiemwen Street, off Wire Road, Benin City.	White solid, hygroscopic. Melting point (85.1^{o}C), decomposes 100^{o}C), density (2.54g/cm^3) Provides an accurate pH of washing water.
Calcium Hypochlorite	Juliros Nigeria Enterprises, 21 B, Evbiemwen Street, off Wire Road, Benin City.	A white/gray powder. Melting point (100^{o}C), decomposes (175^{o}C), density (2.35g/cm^3) $Ca(ClO_2)$ Softener and bleach.
Hydrogen Peroxide	Juliros Nigeria Enterprises, 21 B, Evbiemwen Street, off Wire Road, Benin City.	A very pale blue liquid slightly viscous than water, colourless in water. Melting point (-0.43^{o}C), boiling point (150^{o}C), density(1.463g/cm^3) Bleaching agent.
n-hexane	Juliros Nigeria Enterprises, 21 B, Evbiemwen Street, off Wire Road, Benin City.	A liquid solvent. Melting point (69^{o}C), boiling point (-95^{o}C), density (0.66g/cm^3)
Sodium Chloride	Juliros Nigeria Enterprises, 21 B, Evbiemwen Street, off Wire Road, Benin City.	Colourless/white crystalline solid, Melting point (801^{o}C), boiling point (1413^{o}C), density (2.165g/cm^3) Enhances formation of granules.
Hard Water	Okpella, Edo State	Water With High Mineral Content. Ph (8.4), Hardness (142mg/l of $CaCO_3$ in water)
Linear Alkyl Benzene Sulfonate	Juliros Nigeria Enterprises, 21 B, Evbiemwen Street, off Wire Road, Benin City.	A liquid, melting point (277^{o}C), boiling point (637^{o}C), density 1.06g/cm^3), pH (7-10) in 1% water solution.

4.0 Production of Detergent

Formulations in Table 2 were used to produce the detergents from the unmodified rubber and cherry seed oils, LABS, esterified and benzene-modified rubber and cherry seed oils. The oils used are: unmodified cherry seed oil (formulation 1), unmodified rubber seed oil (formulation 2), linear alkyl benzene sulfonate (formulation 3), benzene-modified esterified rubber seed oil (formulation 4), benzene-modified cherry seed oil (formulation 5), and Benzene-modified rubber seed oil (formulations 6–10).

Table 2: Formulations Used in the Production of Detergents from Rubber Seed and Cherry Seed Oils

Additives	Formulation									
	Parts per 100g weight of Seed Oil									
	1	2	3	4	5	6	7	8	9	10
Linear Alkyl Benzene Sulfonate (LABS)	-	-	100	-	-	-	-	-	-	-
Esterified Rubber Seed Oil	-	-	-	100	-	-	-	-	-	-
Cherry Seed Oil	100	-	-	-	100					
Rubber Seed Oil	-	100	-	-	-	100	100	100	100	100
Benzene	-	-	-	34.0	34.0	34.0	42.5	51.0	59.5	68.0
Sulfonic Acid	5	5	5	5	5	5	5	5	5	5
0.1M Sodium Hydroxide	200	200	200	200	200	200	200	200	200	200
Calcium Hypochlorite	10	10	10	10	10	10	10	10	10	10
Sodium Chloride	30	30	30	30	30	30	30	30	30	30
Hydrogen Peroxide	5	5	5	5	5	5	5	5	5	5

0.1M sodium hydroxide solution was prepared by weighing 4g of sodium hydroxide pellets into a beaker containing 1000ml of water and shaken vigorously. This was left for about 24 hours to allow for cooling to room temperature as the reaction was highly exothermic.

100g of the seed oil was weighed and placed into a stainless steel plate into which was placed a stirrer. Approximately 34g of benzene was then weighed and poured into the plate containing the oil and stirred for about 5 minutes (to modify the oil). 5g of the sulfonic acid was added while stirring continued. 200g of 0.1M sodium hydroxide, 10g of calcium hypochlorite, and 30g of sodium chloride were then added and the mixture stirred continuously with a glass rod and the reaction allowed to completion after which 5g of hydrogen peroxide (bleaching agent) was introduced into the reaction mixture.

The sodium hydroxide was added to also reduce the foam formed during the reaction while hydrogen peroxide was added to effect the disintegration of the detergent to fine powder. When the foaming had subsided, the product was then poured and spread on flat platforms to allow for evaporation at room temperature for about 24 hours. The powdered detergent formed was then subjected to various tests and comparisons to access the effectiveness of the process and the detergent properties.

Rubber seed oil in formulation 4 was esterified with methanol, ethanol and propanol respectively. Formulation 6 was used to produce detergents samples from rubber seed oil extracted at different temperatures [A(40^{o}C), B(50^{o}C), C(60^{o}C), D(70^{o}C), and E(90^{o}C)] while formulations 7-10 are 25% step increase in the amount of benzene used for modifying the rubber seed oil up to a maximum of 100%.

5.0 Method of Empirical Analysis

The readings from the various detergent analyses were subjected to inferential statistical analyses using regression model and analysis of variance (ANOVA). The model is used to determine the relationships between parameters of samples measured with time (in hours) on one hand and the comparisons of the samples, with each other. These are basically to know the degree of association of time and the parameters. The ANOVA technique is adopted to test for significance of the different detergent analyses using various samples and temperatures as discussed below.

5.1 Results and Discussion of Statistical Analyses of Detergent Results

A two-way analysis of variance is the statistical technique adopted to analyse the experimental result of the sample tested. It addresess if there are: (a) there a significant difference between the groups? (b) If so, which groups are significantly different from each other? Statistical tests are provided to compare group means, group medians, and group standard deviations. When comparing means, multiple range tests are used [(7)].

6.0 Test of Hypothesis and Findings

Ho: There is no significant difference among the variation of pH values of detergents solution.

Table 3Tests of Between-Subjects Effects

Dependent Variable: pH Values with Detergent Sample

Source	Sum of Squares	Df	Mean Square	F	Sig.
SAMPLE	4.031	10	.403	14.824	.000*
TEMP	.095	5	.019	.701	.625
Error	1.360	50	.027		
Total	5.486	65			

a. R Squared = .752 (Adjusted R Squared = .678) * Significant at 5%.

pH Analysis (ANOVA) Result Output of SPSS Version 17.0.

Fcalculated= Mean Sum of Squares Sample/Mean of Squares Error

Fcal.(sample)= 0.403/0.027

=14.824 p.value=0.000*

Fcalculated= Mean Sum of Squares Sample/Mean of Squares Error

Fcal.(Temp)= 0.019/0.027

=0.701 p.value=0.625

6.1 Decision

If P-value>0.05, accept Ho otherwise P-value<0.05, accept H1. Based on these criteria, the ANOVA result showed that the samples are statistically significant to the OMO but not significant to temperatures with regards to pHvalue. These are evident from the values of P less than 5% critical value for samples but greater than 5% level in temperature variation during the experiment.

Table 4: Multiple Comparisons

pH Values with Detergent Sample

LSD

(I) Sample	(J) Sample	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
OMO	sample1	-.0833	.09521	.386	-.2746	.1079
	Sample2	-.3500*	.09521	.001	-.5412	-.1588
	sample3	-.5667*	.09521	.000	-.7579	-.3754
	sample4	-.6500*	.09521	.000	-.8412	-.4588
	sample5	-.3833*	.09521	.000	-.5746	-.1921
	sample6	-.5500*	.09521	.000	-.7412	-.3588
	Sample7	-.6333*	.09521	.000	-.8246	-.4421
	Sample8	-.7000*	.09521	.000	-.8912	-.5088
	Sample9	-.7333*	.09521	.000	-.9246	-.5421
	Sample10	-.7667*	.09521	.000	-.9579	-.5754
sample1	OMO	.0833	.09521	.386	-.1079	.2746
	sample2	-.2667*	.09521	.007	-.4579	-.0754
	sample3	-.4833*	.09521	.000	-.6746	-.2921
	sample4	-.5667*	.09521	.000	-.7579	-.3754
	sample5	-.3000*	.09521	.003	-.4912	-.1088
	sample6	-.4667*	.09521	.000	-.6579	-.2754
	sample7	-.5500*	.09521	.000	-.7412	-.3588
	sample8	-.6167*	.09521	.000	-.8079	-.4254
	sample9	-.6500*	.09521	.000	-.8412	-.4588
	Sample10	-.6833*	.09521	.000	-.8746	-.4921
sample2	OMO	.3500*	.09521	.001	.1588	.5412

	sample1	.2667*	.09521	.007	.0754	.4579
	sample3	-.2167*	.09521	.027	-.4079	-.0254
	sample4	-.3000*	.09521	.003	-.4912	-.1088
	sample5	-.0333	.09521	.728	-.2246	.1579
	sample6	-.2000*	.09521	.041	-.3912	-.0088
	sample7	-.2833*	.09521	.004	-.4746	-.0921
	sample8	-.3500*	.09521	.001	-.5412	-.1588
	sample9	-.3833*	.09521	.000	-.5746	-.1921
	sample10	-.4167*	.09521	.000	-.6079	-.2254
sample3	OMO	.5667*	.09521	.000	.3754	.7579
	sample1	.4833*	.09521	.000	.2921	.6746
	sample2	.2167*	.09521	.027	.0254	.4079
	Sample4	-.0833	.09521	.386	-.2746	.1079
	Sample5	.1833	.09521	.060	-.0079	.3746
	Sample6	.0167	.09521	.862	-.1746	.2079
	Sample7	-.0667	.09521	.487	-.2579	.1246
	Sample8	-.1333	.09521	.168	-.3246	.0579
	Sample9	-.1667	.09521	.086	-.3579	.0246
	Sample10	-.2000*	.09521	.041	-.3912	-.0088
sample4	OMO	.6500*	.09521	.000	.4588	.8412
	sample1	.5667*	.09521	.000	.3754	.7579
	sample2	.3000*	.09521	.003	.1088	.4912
	sample3	.0833	.09521	.386	-.1079	.2746
	sample5	.2667*	.09521	.007	.0754	.4579
	sample6	.1000	.09521	.299	-.0912	.2912
	sample7	.0167	.09521	.862	-.1746	.2079
	Sample8	-.0500	.09521	.602	-.2412	.1412
	Sample9	-.0833	.09521	.386	-.2746	.1079
	Sample10	-.1167	.09521	.226	-.3079	.0746
sample5	OMO	.3833*	.09521	.000	.1921	.5746
	sample1	.3000*	.09521	.003	.1088	.4912
	sample2	.0333	.09521	.728	-.1579	.2246
	sample3	-.1833	.09521	.060	-.3746	.0079
	sample4	-.2667*	.09521	.007	-.4579	-.0754
	sample6	-.1667	.09521	.086	-.3579	.0246
	sample7	-.2500*	.09521	.011	-.4412	-.0588
	sample8	-.3167*	.09521	.002	-.5079	-.1254
	sample9	-.3500*	.09521	.001	-.5412	-.1588
	sample10	-.3833*	.09521	.000	-.5746	-.1921
sample6	OMO	.5500*	.09521	.000	.3588	.7412
	Sample1	.4667*	.09521	.000	.2754	.6579
	Sample2	.2000*	.09521	.041	.0088	.3912
	Sample3	-.0167	.09521	.862	-.2079	.1746
	Sample4	-.1000	.09521	.299	-.2912	.0912
	Sample5	.1667	.09521	.086	-.0246	.3579
	Sample7	-.0833	.09521	.386	-.2746	.1079
	Sample8	-.1500	.09521	.121	-.3412	.0412

	Sample9	-.1833	.09521	.060	-.3746	.0079
	Sample10	-.2167*	.09521	.027	-.4079	-.0254
sample7	OMO	.6333*	.09521	.000	.4421	.8246
	sample1	.5500*	.09521	.000	.3588	.7412
	sample2	.2833*	.09521	.004	.0921	.4746
	sample3	.0667	.09521	.487	-.1246	.2579
	sample4	-.0167	.09521	.862	-.2079	.1746
	Sample5	.2500*	.09521	.011	.0588	.4412
	Sample6	.0833	.09521	.386	-.1079	.2746
	Sample8	-.0667	.09521	.487	-.2579	.1246
	Sample9	-.1000	.09521	.299	-.2912	.0912
	Sample10	-.1333	.09521	.168	-.3246	.0579
sample8	OMO	.7000*	.09521	.000	.5088	.8912
	sample1	.6167*	.09521	.000	.4254	.8079
	sample2	.3500*	.09521	.001	.1588	.5412
	sample3	.1333	.09521	.168	-.0579	.3246
	sample4	.0500	.09521	.602	-.1412	.2412
	sample5	.3167*	.09521	.002	.1254	.5079
	sample6	.1500	.09521	.121	-.0412	.3412
	sample7	.0667	.09521	.487	-.1246	.2579
	sample9	-.0333	.09521	.728	-.2246	.1579
	Sample10	-.0667	.09521	.487	-.2579	.1246
sample9	OMO	.7333*	.09521	.000	.5421	.9246
	sample1	.6500*	.09521	.000	.4588	.8412
	sample2	.3833*	.09521	.000	.1921	.5746
	sample3	.1667	.09521	.086	-.0246	.3579
	sample4	.0833	.09521	.386	-.1079	.2746
	sample5	.3500*	.09521	.001	.1588	.5412
	sample6	.1833	.09521	.060	-.0079	.3746
	sample7	.1000	.09521	.299	-.0912	.2912
	sample8	.0333	.09521	.728	-.1579	.2246
	sample10	-.0333	.09521	.728	-.2246	.1579
sample10	OMO	.7667*	.09521	.000	.5754	.9579
	sample1	.6833*	.09521	.000	.4921	.8746
	sample2	.4167*	.09521	.000	.2254	.6079
	Sample3	.2000*	.09521	.041	.0088	.3912
	Sample4	.1167	.09521	.226	-.0746	.3079
	Sample5	.3833*	.09521	.000	.1921	.5746
	Sample6	.2167*	.09521	.027	.0254	.4079
	Sample7	.1333	.09521	.168	-.0579	.3246
	Sample8	.0667	.09521	.487	-.1246	.2579
	Sample9	.0333	.09521	.728	-.1579	.2246

Based on observed means.The error term is Mean Square(Error) = .027.

*. The mean difference is significant at the 0.05 level.

Conclusion

Screening of various seed oils for suitability as potential component in detergent formulation and production of

detergents using neat, benzene-modified, and esterified rubber seed oils. Assessment of the properties such as pH, lathering ability and cleansing power of the produced detergent. From the result of ANOVA of the SPSS version 17.0 result output, the p-value of the samples of the experimental procedure is statistically significant as the p-value (0.000) is less than the critical value of 0.05. The Duncan Multiple Range Test statistic revealed the actual existence of significance using the indicator of *. Omo was found to be difference in the assessment of detergent in the seeds oil based on the varying temperature with sample 2 through to10 expect sample 1 which is not statistically significant to Omo. This implies that omo has the same cleaning ability with other sample 1 based on the quality and characteristics experiment but different from samples2 through to10.

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